TECHNICAL UNIVERSITY OF LIBEREC

FACULTY OF TEXTILE ENGINEERING

DIPLOMA THESIS

2012 NOKUTHULA ADELAIDE SHEZI

FACULTY OF TEXTILE ENGINEERING

DEPARTMENT OF TEXTILE CHEMISTRY

Carboxymethylcellulose on fibers

Nokuthula Adelaide Shezi

Supervisor : Ing. Karolína Borůvková

Consultant: doc. Ing. Jakub Wiener, Ph.D.

Number of pages: 93

Number of tables: 24

Number of figures: 14

Number of graphs: 18

Number of appendices: 3

Statement

I have been informed that on my thesis is fully applicable the Act No. 121/2000 Coll. about copyright, especially §60 - school work.

I acknowledge that Technical University of Liberec (TUL) does not breach my copyright when using my thesis for internal need of TUL.

Shall I use my thesis or shall I award a licence for its utilisation I acknowledge that I am obliged to inform TUL about this fact, TUL has right to claim expenses incurred for this thesis up to amount of actual full expenses.

I have elaborated on the thesis alone utilising listed literature and on basis of consultations with the supervisor.

Date: 9 May 2012

Signature: Nokuthula Adelaide Shezi

Acknowledgement

I would like to pass my sincere gratitude to my supervisor Ing. Karolina Boruvkova for her support and professional help to make this work successful. I would also like to thank doc. Ing. Jakub Wiener Ph.D. sincerely who helped me to accomplish the task to conclude this project as well as the laboratory technicians and the Technical University of Liberec academics and staff ,your efforts and support were highly appeciated and I will be forever grateful to you, and above all I give my honour and praise to God for guiding me all the way.

I would also like to thank the KwaZulu Natal Department of Economic Development and Tourism (South Africa) for their financial support during my study in Czech Republic.

Abstract

Carboxymethylcellulose salt occurs as a white or slight yellow flocculent fiber powder or white powder which is non-toxic, tasteless, odorless and easy to dissolve. It has a thickening, emulsifying, shaping, water retention, stabilizing capacity, and becomes colourless when dissolved in cold or hot water. Carboxymethylcellulose has found a wide use in textile industry for textile surface treatment. It has gained an advantage over starch as a sizing agent for cotton yarn since it enables a smooth surface.

This work discusses the use of CMC to modify the surface of polyester fibers.The topochemical modified polyester fibers are improved by the the crosslinking of polyester fibers with carboxymethylcellulose resulting in a better water absorption capacity of the polyester fibers since the polyester fibers are well known for their hydrophobic character.

The overall objective of this research is to observe and determine the best crosslinking temparature and crosslinking time of the carboxymethylcellulose onto the polyester textile substrate and also to observe the maximum water absorption capacity of the treated fabric. The oven as well as the special heating vials with lids were used to perfom the whole experiments to try and accomplish the whole task of this research.

Table of contents

List of Abbreviations

CMC	Carboxymethylcellulose Salt
% CM	Carboxymethyl content
DS	Degree of Substitution
DP	Degree of Polymerization
PES	Polyester
MW	Molecular Weight
N	Normality
M	Molar
m	Mass
Cu	Copper
Pb	Lead
Sn	Tin
BOD	Biochemical Oxygen Demand

1 Introduction

The various properties of CMC depend upon three factors ie. molecular weight of the polymer, average number of carboxyl content per anhydroglucose unit, and the distribution of carboxyl substituents along the polymer chains. The most important properties of CMC are viscosity building and flocculation. Among all the polysaccharides, CMC is easily available and it is also very cheap. It has high shear stability. [1]

Recent developments in textile industry have found the use of carboxymethylcellulose as a good alternative over the use of starch as a sizing agent for cotton since dyeing can be done without desizing due to the rheological properties of carboxymethylcellulose. An increase in stiffness of cotton has been noted with the use of a solution CMC as a sizing agent over starch. [2]

Along with the increase in stiffness in textiles sized with a solution of CMC other desirable properties were also enhanced such as lack of stickiness during drying and ironing a lack of staining of coloured textiles and a superior feeling of softness compared to textiles sized with starch. A further desirable characteristic of textiles sized with a solution of CMC that is observed is that of excellent resilient properties. [3]

It has also been discovered that a solution of CMC can be used to modify the surfaces of textiles. [4]

Polyester fiber has a highly compact and crystalline structure, and is markedly hydrophobic. CMC absorbs moisture from the atmosphere. The amount of moisture absorbed depend on the initial moisture content and on the relative humidity and temperature of the surrounding air. [5]

Carboxymethylcellulose can be crosslinked with polyester thermally to improve the surface properties of polyester and improve its water absorption capacity. This could be easily demonstrated on the construction of a tampon that is used for sanitary purposes. [6]

Instead of using the cotton and rayon in the manufacturing of a tampon, carboxymethylcellulose and compressed beads of polyester can be used for maximum

absorption. Carboxymethylcellulose can provide a comfortable vaginal dryness feeling by absorbing the natural humidity of the vagina. [7]

In this research we will try to observe the best crosslinking temperature and time of the solution of CMC to the polyester substrate. Also we would try to compare the absorption capacity of the treated fabric to that of the untreated fabric. Different types of carboxymethyl salts were used to obtain comparable results.

A chemical crosslinking agent , epichlorohydrin was used to crosslink CMC to compare the results obtained with that of the thermally crosslinked samples. Epichlorohydrin is preferably used because it achieves simultaneous crosslinking as well as etherification. [8]

This process is for the production of a water adsorbing cellulose ether. Cellulose ethers produced and modified can absorb water very rapidly. [9]

2 Literature review

2.1 Carboxymethylcellulose description

Fig. 1: Carboxymethylcellulose Salt

Appearance is white or slight yellow flocculent fiber powder or white powder, non-toxic, tasteless, odorless and easy to dissolve. Has thickening, emulsifying, shaping, water retention, stabilizing capacity, and becomes colourless when dissolved in cold or hot water. It is well known that cellulose is not water soluble, but CMC is made water soluble as a result of a chemical reaction between cellulose and monochloroacetic acid (MCA) in the presence of sodium hydroxide. [10]
CMC can form high viscosity colloid solution, there is adhesion, thickening, mobility, emulsifying, shaphing, water, protective colloid, film forming, acid, salt, and other features for suspension, and no physical damage, so in food, medicine, daily chemical, petroleum, paper, textiles, construction and other fields is widely used in production.[11]

Sodium carboxymethylcellulose (CMC) are anionic cellulose ethers, look for white or light yellow flocculent fiber powder or white powder, odorless and tasteless, non-toxic, soluble in cold water or hot water, the formation of a certain stick degree of transparent solution. Is neutral or slightly alkaline solution, insoluble in ethanol, ethyl ether, isopropyl alcohol, acetone, and other organic solvents, soluble in water 60% ethanol or acetone solution. Hygroscopicity, light thermal stability, viscosity decreases with increasing temperature, the solution pH value of 2 to 10 in the stable, pH less than 2, solid precipitation, pH value of more than 10 viscosity. Color

temperature of 227 °C, charring temperature of 252 °C, 2% solution surface tension 71 mn/n.[9]

2.1.1 Properties and Structure of Carboxymethylcellulose

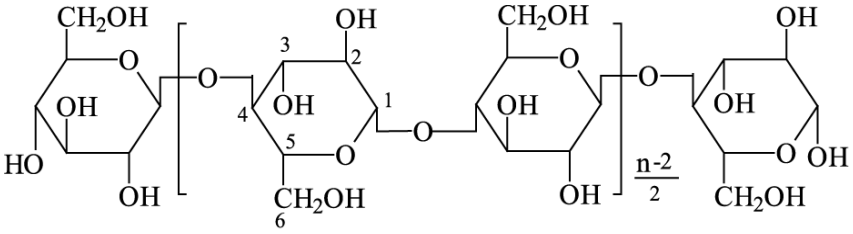


Fig.2: Structure of Cellulose

Table 1 : Physical properties of CMC

Property	
Appearance	White or slight yellow flocculent powder.
Density	1500 kg/m^3
Melting point	300 °C
Water Solubility	Soluble
Solvent solubility	Insoluble in acetone
Chemical Formula	(C35H49O29)n
Stability	Stable under normal conditions

Figure 2 shows the structure of the cellulose molecule. It is visualized as a polymer chain composed of repeating cellobiose units (in brackets). These, in turn, are composed of two anhydroglucose units (ß-glucopyranose residues). In this structure, n is the number of anhydroglucose units (which are joined through 1,4 glucosidic linkages) or the degree of polymerization, of celulose. [8]

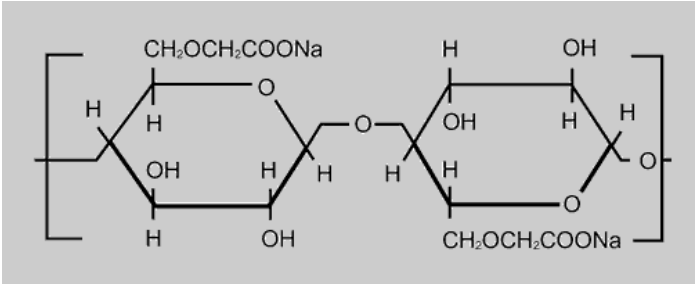


Fig. 3: Idealized Structure of Carboxymethylcellulose

CMC is a cellulose ether that is produced by reacting alkali cellulose with sodium monochloroacetate under rigidly controlled conditions. Cellulose ethers, such as CMC, are long-chain polymers. [8]

2.1.2 Uses of Carboxymethylcellulose in Textile Industry

- Laundry and Fabric Sizes Film former. - Instead of starch.

CMC is soluble in cold water and does not require a cooking step . Solutions remain fluid a t room temperature and don't retrograde. They can be reheated and cooled repeatedly. It is easy to remove and redissolve CMC size in warm water CMC like starch supports mildew on storage of fabrics. Because of its film forming properties it is used in the protective coating field where it serves as a grease-resistant sealing coat for walls and other porous surfaces. CMC is more expensive than starch. [10]

The use of carboxymethylcellulose as a good alternative over the use of starch as a sizing agent for cotton since dyeing can be done without desizing due to the rheological properties of carboxymethylcellulose. An increase in stiffness of cotton has been noted with the use of a solution CMC as a sizing agent over starch. Along with the increase in stiffness in textiles sized with a solution of CMC other desirable properties were also enhanced such as lack of stickiness during drying and ironing, a lack of staining of coloured textiles and a superior feeling of softness compared to textiles sized with starch. A further desirable characteristic of textiles sized with a solution of CMC that is observed is that of excellent resilient properties. [11]

- Latex Adhesives – Rheology control thickener, water binding and holdout.

Most of the CMC applications are based on its rheological properties. The ability of CMC to function as a thickener or flow-control agent depends largely on its degree of substitution and on the molecular weight, but also on the relative stiffness of the cellulosic backbone. [5]

- Printing Pastes and Dyes – Thickener, water binding.

Used in textile industry as a thickner in printing industry.reactive dyes contain reactive groups which under dyeing conditions react with substrate under alkaline

conditions and fix the dye by covalent bonding.thickners which are similar in structure to the substrate to dyed are normally unsuitable because they are capable of reacting with reactive dyes leading to poor dye yields.most thickners used are alginates and cmc based thickners are easily removed by washing,alginates at higher ph values trans eliminative depolymerisation is observed .alginate areeasily degradable by mico organisms. [11]

- Warp Sizing-High film strength, good adhesion to fiber, low BOD value.

Carboxymethylcellulose salts possess greater utility as sizing and finishing agents. Aqueous solutions of the sodium salts of CMC are particularly useful for warp sizing of yarns filaments and threads. [11]

2.2 Crosslinking

Crosslinking can be accomplished chemically or by irradiation. Chemical crosslinking with rubber material is called Vulcanization. It is accomplished by a heat induced reaction between the polymers and a crosslinking agent. For wire and cable insulations, chemical crosslinking is performed by passing the wire through a long pressurized steam tube called a continuous vulcanizing. [12]

Carboxymethyl cellulose is a biocompatible macromolecule that has been used for drug delivery systems in many investigations with the aim of controlled drug release with various ways: it has been used for a microgel matrix for a new type of sticking plaster; due to its adhesive characteristic it has been used as bioadhesive material.[13]

Polymer modification is required to bring specific properties to the modified material, such as enhanced thermal stability, multiphase physical responses, compatibility, impact response, flexibility, and rigidity. Modifications make an insoluble polymer from a soluble one or vice versa. Thus polymer modification improves the processibility of the polymers. One of the recent directions regarding polymer modification is intended to avoid their nonbiodegradable nature. [12]

- Cross-Linking Provides:

1. Higher tensile strength
2. Improved abrasion/cut through
7. Improved fluid resistance
8. Slightly better flame resistance

3. Better crush resistance
4. Solder iron resistance
5. Better over load characteristics
6. Resistance to stress cracking
9. No change of electricals
10. Negligible change in thermal stability
11. Decrease in flexibility
12. Improved high temperature mechanicals

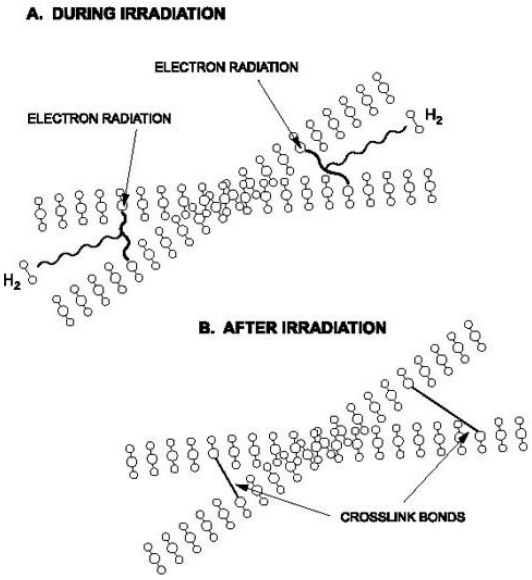


Fig. 4: Structure of crosslinking bonds before and after radiation

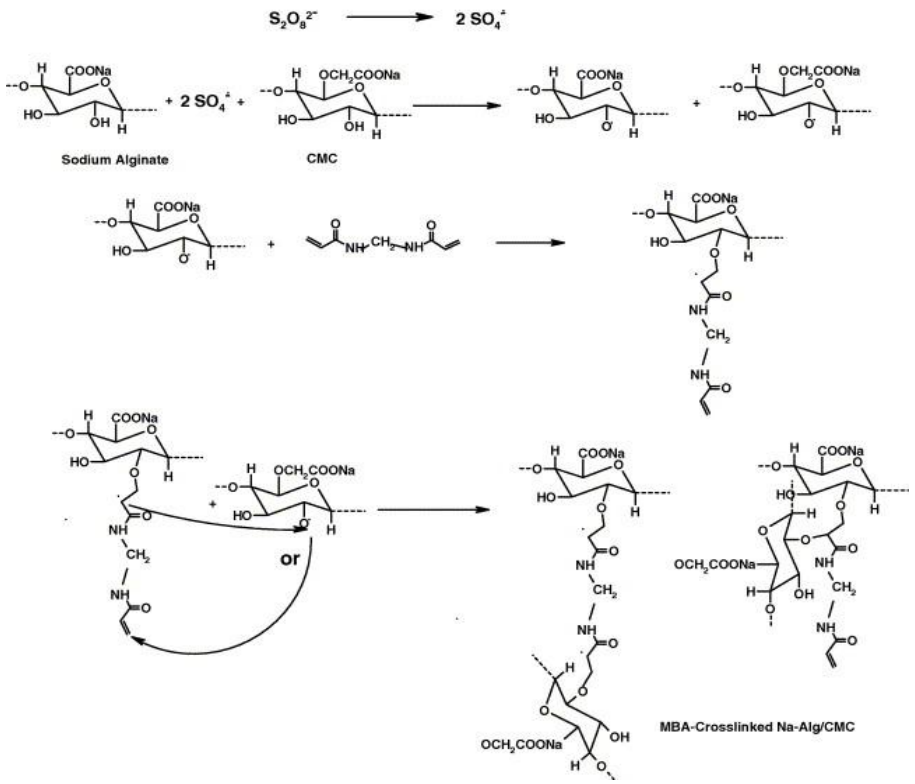


Fig. 5: Mechanism of covalent cross-linking of carboxymethyl chitosan (CMC) [11]

2.3 Degree of Substitution

The term degree of substitution refers to the average number of the carboxymethyl groups substituted on the anhydroglucose unit. The functional properties of CMC depend on the degree of substitution of the cellulose structure (i.e., how many of the hydroxyl groups have taken part in the substitution reaction), and also on the chain length of the cellulose backbone. The degree of substitution (DS) of CMC is usually in the range 0.6 to 0.95 derivatives per monomer unit.CMC is generally considered a water-soluble polymer down to DS 0.3. [9]

Only those sodium carboxymethylcellulose which have a degree of substitution of 2 or higher can at present be used as the only thickeners and flow improvers for reactive printing. The reason why CMC with a lower degree of etherification cannot be used lies in the hardening of the handle of the textile fabric which occurs after printing due to reactions of the CMC with the dye via remaining OH groups. [9]

The reason why a CMC having a degree of substitution below 2 can still react with the reactive dye lies in the structure of CMC and the types of substitution of the carboxymethyl groups within an anhydroglucose unit.

Compared withcarboxymethylcellulose, alginate, whose C.sub.6 -position is a carboxyl function so that it has a degree of substitution of carboxyl functions of 1,generally does not react with reactive dyes. [11]

According to the state of the art, therefore, the disadvantages of the hardening of the handle in reactive printing due to the use of CMC as thickener can only be overcome with a degree of substitution above 2.2. [9]

The preparation of a CMC having a DS above 2.2 is, however, cost intensive. Firstly, the yield of reagent decreases with increasing degree of substitution and secondly the salt produced in the reaction leads to low viscosity stages unless itis removed from the CMC by expensive processes. [5]

The technical problem therefore arose of providing a carboxymethylcellulose which does not produce a reaction with the reactive dye and hence hardening of the handle of the substrate (textile fabric) even at degrees of substitution below 2.2. Such a CMC should be comparable with alginate in its yield and its properties in printing. The

effect of the Degree of Substitution on the properties of CMC is shown below Tolerance to salt increases and tendency towards thixotropic behavior decreases as the degree of substitution increases. Thixotropy decreases as the degree of substitution of the CMC increases. [5]

The degree of substitution is a very important parameter affecting water retention capacity of crosslinked CMC absorbents. There is another factor which is as important as the Degree of Substitution, and that is the "Uniformity of Substitution".

The "smooth" or non-substituted regions of a non-uniformly substituted molecule behave just like cellulose because they are still cellulose! These regions can hydrogen bond to a similar region on an adjacent molecule, leading to the build up of a loose gel network. This build up is time dependent, and is called "thixotropy". [8]

The loose gel network can be disrupted by shearing the CMC solution, but upon standing under no shear conditions the network will reform over time. Solutions of CMC can be prepared in a wide range of viscosities. Such solutions are non Newtonian because they change in viscosity with the change in shear rate. Consequently it is essential to standardise viscosity determination methods. This standardisation must include the type and extent of agitation used to dissolve the CMC as well as the precise control of temperature, conditions of shear and method of viscosity measurement. [8]

The viscosity of aqueous solutions increases sharply with an increase in concentration. If a high viscosity CMC appears to a viscous syrup when it is poured from the bottle it behaves as a thin liquid when applied as a lotion and yet when high shear stress is removed it will instantly revert to its original highly viscous state. This type of behaviour is referred to as Pseudoplasticity or time-independent shear thinning, a form of non-Newtonian flow. It differs from the time dependent viscosity change called Thixotropy. [8]

A non-Newtonian fluid is a fluid whose flow properties differ in any way from those of Newtonian fluids. Most commonly the viscosity of non-Newtonian fluids is not independent of shear rate or shear rate history. However, there are some non-Newtonian fluids with shear-independent viscosity, that nonetheless exhibit normal stress-differences or other non-Newtonian behavior. Many salt solutions and molten polymers are non-Newtonian fluids.

In a non-Newtonian fluid, the relation between the shear stress and the shear rate is different, and can even be time-dependent. Therefore a constant coefficient of viscosity cannot be defined.

Although the concept of viscosity is commonly used to characterize a material, it can be inadequate to describe the mechanical behavior of a substance, particularly non-Newtonian fluids.

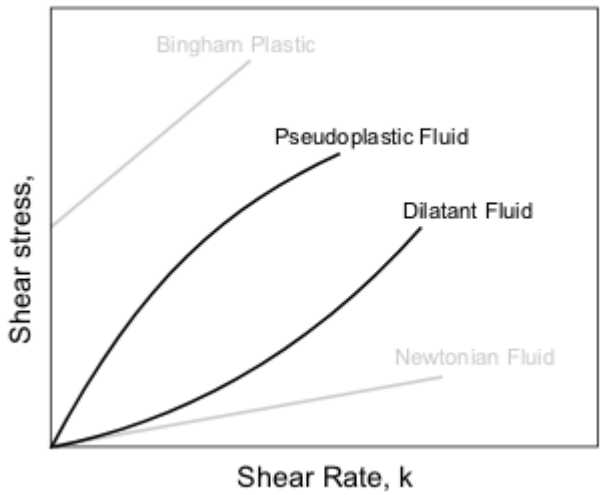


Fig. 6: Pseudoplasticity Behaviour of Non-Newtonian Fluids

2.4 CMC solutions

CMC solutions in the concentration range 1–5% and have reported nearly Newtonian behavior at the lowest concentration and pseudoplastic,thixotropic, and viscoelastic responses at the higher-endconcentrations. [9]

At higher CMC concentrations, above 1%, the viscosity curves are quiet different, i.e., an initial shear-thickening behavior is observed, where the apparent viscosity increases with increasing shear rate, followed at a given shear rate by a shear-thinning behavior. Similar observations reported Newtonian or shear-thinning behavior of polymer solutions at low shear rates and shear-thickening behavior when the shear rate is increased above a critical value. Moreover, when the shear rate is further increased, shear-thinning behavior is observed. In practice, this means that the flow encounters less resistance at higher shear rates.It is admitted that this shear-thinning

behavior is caused by the disentanglement of the polymer coils in solution or increased orientation of the polymer coils in the direction of flow. [5][8]

For all CMC concentrations, a decrease of the elastic compliance (or an increase of the elastic modulus E =1 /J),with the increase in concentration is observed. This indicates that the creep deformation decreases with increasing the polymer concentration and the time necessary to reach a constant deformation during recovery, after removal of the shear stress decreases. This is characteristic of systems whose links in the inner structure strengthen when the concentration is increased, in agreement with the shear flow. [11]

Solutions of all CMC solutions display pseudoplastic behavior. The solutions with higher molecular weight and low degree of substitution normally shows thixotropic behavior in solution which is best explained by high viscosity. [5][8]

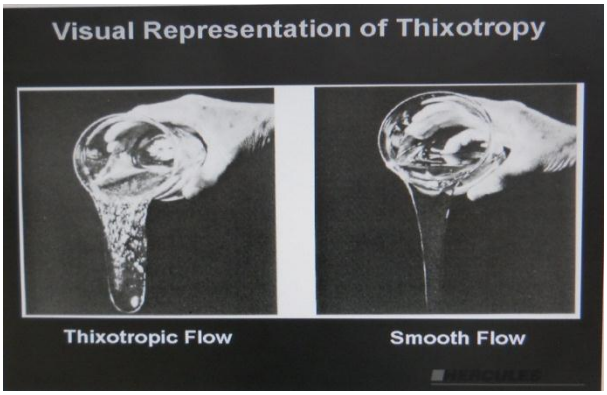


Fig. 7: Shows Smooth Flow to Thixotropic Flow of CMC Solutions

2.5 Polyester Fibers

Polyester fiber is a " manufactured fiber in which the fiber forming substance is any long chain synthetic polymer composed at least 85% by weight of an ester of a dihydric alcohol (HOROH) and terephthalic acid (p-HOOC-C_6H_4COOH)". The most widely used polyester fiber is made from the linear polymer poly (ethylene terephtalate), and this polyester class is generally referred to simply as PET. High strength, high modulus, low _shrinkage, heat set stability, light fastness and chemical resistance account for the great versatility of PET. [15]

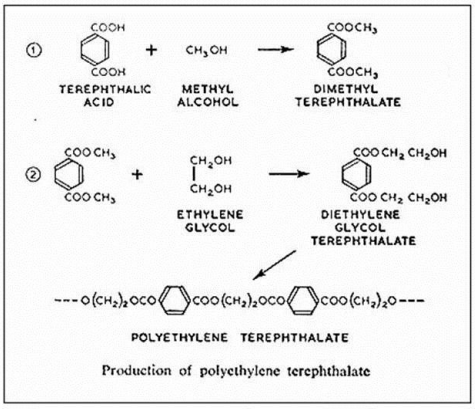


Fig. 8: Production of Polyester [15]

Table 2: Properties of Polyester Fibers [15]

	Filament yarn		Staple and tow	
Property	Regular tenacity[a]	High tenacity[b]	Regular tenacity[c]	High tenacity[d]
breaking tenacity,[e] N/tex	0.35-0.5	0.62-0.85	0.35-0.47	0.48-0.61
breaking elongation	24-50	10-20	35-60	17-40
elastic recovery at 5% elongation, %	88-93	90	75-85	75-85
initial modulus, N/tex[f]	6.6-8.8	10.2-10.6	2.2-3.5	4.0-4.9
specific gravity	1.38	1.39	1.38	1.38
Moisture regian, %	0.4	0.4	0.4	0.4
Melting temperature, ^{o}C	258-263	258-263	258-263	258-263

a Textile-filament yarns for woven and knit fabrics.

b Tire cord and high strength, high modulus industrial yarns.

c Regular staple for 100% polyester fabrics, carpet yarn, fiberfill, and blends with cellulosic blends or wool.

d High strength, high modulus staple for industrial applications, sewing thread and cellulosic blends.

e Standard measurements are conducted in air at 65% rh and 22^{o}C.

f To convert N/text to ge/den, multiply by 11.33.

g The equilibrim moisure content of the fibers at 21^{o}C and 65% rh.[15]

2.5.1 Characteristics of polyester

- Polyester fabrics and fibers are extremely strong.
- Polyester is very durable: resistant to most chemicals, stretching and shrinking, wrinkle resistant, mildew and abrasion resistant.
- Polyester is hydrophobic in nature and quick drying. It can be used for insulation by manufacturing hollow fibers.
- Polyester retains its shape and hence is good for making outdoor clothing for harsh climates.
- It is easily washed and dried.[15]

2.5.2 PES nonvowen fabric

Nonwoven structure of PES was used in the experiment. The cost of polyester, with the combination of its superior strength and resilience, is lower than that of rayon. Polyester fibers are hydrophobic, which is desirable for lightweight facing fabrics used in the disposable industry. They provide a perceptible dry feel on the facing, even when the inner absorbent media is saturated. As new methods of processing and bonding of PET are developed, rayon is being replaced by polyester on the market.

Properties of polyester fibers are strongly affected by fiber structure. The fiber structure, which has a strong influence on the applicability of the fiber, depends heavily on the process parameters of fiber formation such as spinning speed (threadlike stress), hot drawing (stretching), stress relaxation and heat setting (stabilization) speed. [3]

As the stress in the spinning threadlike is increased by higher wind-up speed, the PET molecules are extended, resulting in better as-spun uniformity, lower elongation and higher strength, greater orientation and high crystallinity. Hot drawing accomplishes the same effect and allows even higher degrees of orientation and crystallinity. Relaxation is the releasing of strains and stresses of the extended molecules, which results in reduced shrinkage in drawn fibers.

Heat stabilization is the treatment to "set" the molecular structure, enabling the fibers to resist further dimensional changes. Final fiber structure depends considerably on the temperature, rate of stretching; draw ratio (degree of stretch), relaxation ratio and

heat setting condition. The crystalline and noncrystalline orientation and the percentage of crystallinity can be adjusted significantly in response to these process parameters.

Mechanical properties of nonwoven fabrics depend on many parameters, including fiber properties, web structure and processing. It is, therefore, useful to review some of the elementary knowledge of fiber properties and other factors like web processing techniques and structure. [3]

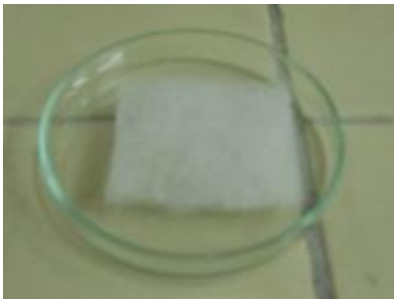


Fig. 9: Nonwoven Sample of Polyester

2.5.3 Epichlorhydrin

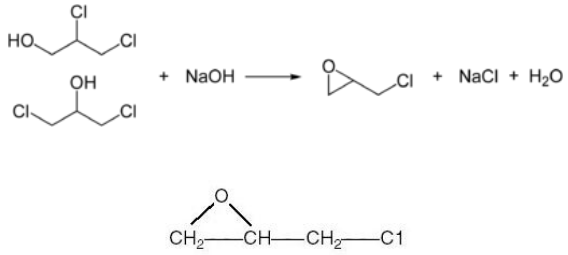


Fig. 10: Chemical structure of epichlorhydrin

Table 3: Physical Properties of Epichlorohydrin

Property	
Appearance	Clear colourless liquid
Melting point	-57.1 °C
Molecular weight	92.53 g/mol
Chemical name	3-Chlorohydrin-1, 2-epoxy propane
Density	1.178 g/cm^3
Viscosity	1.086 mPa.s, 25 °C
Boiling point	116.4 °C

Epichlorohydrin (abbreviated ECH) is an organochlorine compound and an epoxide. This is a colorless liquid with a pungent, garlic-like odor, moderately soluble in water,

but miscible with most polar organic solvents. In its pure form, epichlorohydrin has the presence of both an epoxide ring and a chlorine atom in the molecule allows epichlorohydrin to readily undergo a variety of chemical reactions with many types of compounds. This versatility earns its wide use as a chemical crosslinking agent. At high temperatures, epichlorohydrin can react violently and in some cases explosively (spontaneous polymerization) in the presence of certain substances. [7]

Superabsorbent hydrogels were prepared successfully from carboxymethylcellulose sodium (CMC) and cellulose in the NaOH aqueous system by using epichlorohydrin (ECH) as cross-linker. Superabsorbent hydrogels are three-dimensional cross-linked hydrophilic, linear or branched polymers with the ability to absorb large quantities of water, or physiological solutions compared with general absorbing materials. Because of their excellent hydrophilic properties, high swelling ratio, and biocompatibility, hydrogels are been widely used. [15]

Cellulose and sodium carboxymethylcellulose (CMC) are biocompatible and biodegradable, so they are often used in the biomedical field. Recently, cellulose-based superabsorbent hydrogels prepared by using radiation-induced cross-linking . the application of superabsorbent hydrogels which possess high adsorbent capability, biocompatibility and biodegradability.

CMC was used as hydrophilic filler in the hydrogel network to increase the absorbing of water. The influence of the carboxymethylcellulose composition on the swelling ratio of cellulose/CMC hydrogels in distilled water at room temperature was observed. The samples exhibited high equilibrium swelling ratio, indicating all of the samples were superabsorbent hydrogels. [7]

Superabsorbent hydrogels were fabricated successfully from CMC and cellulose in NaOH aqueous solution by cross-linking with ECH. The superabsorbent mechanism could be described as that the stiff cellulose molecules acted as the strong backbone of the network structure for keeping appearance of the hydrogels including a lot of water, and the highly hydrophilic CMC contributed to the higher swelling ratio. The experimental results proved that the cellulose/CMC hydrogels exhibited superabsorbent capacity and high equilibrium swelling ratio, which could be improved by changing the amount of CMC. The hydrogels possessed release behavior of BSA, and the release time could be controlled by the content of CMC. Their smart swelling,

superabsorbent and controlled release properties will be very important in biomaterials. [1]

2.6 Metal Analysis (analysis of inorganic elements)

The conventional methods used to remove heavy metals include chemical precipitation, ion exchange, electro-dialysis, membrane separations, reverse osmosis and solvent extraction.The search for new, effective and economical technologies involving the removal of toxic metal ions from waste waters has directed attention to biosorption, based on metal binding capacities of various biological materials at little or no cost. [16]

Sodium carboxymethyl cellulose (CMC) is known to be a derivative of cellulose that is colorless, odorless and tasteless, physiologically inert, chemically stable, not dangerous for health and environment and water soluble. It also possesses good complexation ability for some metal ions and is chosen as the macromolecular complexing agent for the selective removal and rejection of copper from water in complexation–microfiltration process. [16]

It is well known that metal ions are too small to be retained by the membrane. In the complexation-microfiltration process heavy metal ions are first complexed by macro ligand in order to increase their molecular weight so having a size larger than pores of selected membrane they can be retained. [16]

2.6.1 Properties of Copper

Copper sulfate is blue and odorless. It gives off moisture when exposed to air. It should not be stored in metal containers. Copper sulfate is also incompatible with acetylene gas and with magnesium metal. It is indefinitely stable when kept dry and is stable to heat, cold, or light. However, there is slight decomposition of copper sulfate at temperatures above 200 °C. Above 400 °C, it decomposes and giving off sulfur trioxide gas. Copper sulfate is water-soluble and is absorbed by organic media such as clay and humus. Copper sulfate melts at temperatures above 100 degrees Celsius and becomes an anhydrous crystal at temperatures above 200 °C. It is not flameable and combustible. [16]

As copper sulfate is a naturally occurring compound, it does not degrade under normal environmental conditions and does not reduce its presence in bodies of water by volatilization. It is strongly reactive with other metals such as steel, iron and magnesium.

2.6.2 Properties of Lead

Lead has the advantages of low melting temperature and extreme malleability, which allow easy casting, shaping and joining of lead articles. Besides this lead is slow to corrode and there are many examples of lead articles which have lasted for centuries. Lead is relatively abundant. Lead concentrates can be easily extracted from the ore and winning the metal from the concentrate does not need much energy. This reflects also in a fairly low price compared with other non-ferrous metals.

Lead can be recycled as a secondary raw material from lead-acid batteries, from metallic scrap and from several composite consumer products in conjunction with existing recycling loops, for example for steel, zinc and copper, at moderate costs. However, compared with other metals, lead has extremely low strength, exacerbated by its creep and fatigue behaviour. [17]

Thus it is unsuitable for applications that require even moderate strength. (Some of its mechanical properties are closer to those of higher strength plastics than most metals.) Lead is rarely used in its pure form, as small alloying additions considerably increase its strength. For applications requiring higher strengths, composites such as lead clad steel can be used. The very high density of lead lends itself to some quite different applications, such as shielding against sound, vibrations and radiation, for example as protection for users of computer and TV screens. For these purposes lead is used in metallic form or as lead compounds in lead glasses. Some compounds of lead have their own useful properties, particularly in relation to colour and glass-forming ability. [17]

It has a very low melting point, compared with most other metals, of 327 °C.Lead has a particularly high density, 11.3g.cm^{-3} compared with many other metals (e.g. iron 7.8g.cm^{-3}, copper 8.9g.cm^{-3}, aluminium 2.7g.cm^{-3}. Lead owes its high density to two factors:

- its high atomic number, and hence high relative atomic mass of 207,

- the metal atoms (or, more precisely, ions) are arranged in a dense, close packed structure (face centred cubic structure). [17]

2.6.3 Properties of Tin

Tin has nearly the whiteness of silver. It is very malleable and flexible, but not elastic. Its tensile strength is too low for it to be drawn into wire. Air will not tarnish it readily, but some acids and strong alkaline solutions attack it noticeably, particularly when hot. [18]

It is a poor conductor of heat and electricity. When pure, a bar or sheet of tin makes a crackling sound when bent, called the "cry of tin"' and as this sound is destroyed by lead as an impurity, this fact is often made use of in testing tin. It melts at about 445 °F. Tin *IS* a chemical element with the symbol Sn (for Latin: *stannum*) and atomic number 50. It is a main group metal in group 14 of the periodic table.

Tin shows chemical similarity to both neighboring group 14 elements, germanium and lead and has two possible oxidation states, +2 and the slightly more stable +4. Tin is the 49th most abundant element and has, with 10 stable isotopes, the largest number of stable isotopes in the periodic table. Tin is obtained chiefly from the mineral cassiterite, where it occurs as tin dioxide, SnO_2.

This silvery, malleable post-transition metal is not easily oxidized in air and is used to coat other metals to prevent corrosion. The first alloy used in large scale since 3000 BC, was bronze, an alloy of tin and copper. [18]

3 Experimental methods and procedures

3.5 Used materials

Different types of CMC were used in this experiment. They were characterized by the difference in molecular weights and the difference in degree of substitution.

Table 4: Types of CMC used in this reasearch work

Carboxymethylcellulose	Appearance	Molecular Weight	Degree of sustitution	Viscosity Type
1.	Off-White	250 000	0.695	Medium
2.	Lt. Brown	250 000	0.7	Medium
3.	Off-White	250 000	1.2	Medium
4.	Off-White	250 000	0.9	Medium
5.	Off-white	700 000	0.9	High
6.	Off-White	90 000	0.7	Low

3.6 Used apparatus

3.6.1 Heated box – Venticell

Due to its patented system of forced air circulation, ensures a homogenous temperature profile for all drying and heating processes. A higher quickness and preciseness of all tempering processes guarantee an economical operation. These units are suitable especially for materials with high humidity.

Fig. 11 The Venticell heated box

3.6.2 Analytical balances – OHAUS ADVENTURE AV213C

Analytical balances are designed for demanding applications, which are capable of measuring to an accuracy of 0.1 mg. It has features enhanced parts counting, dynamic weighing, checkweighing, percentage weighing, totalization, display hold value, determining density or specific gravity by weighing the built-in hook for hanging weighing data GLP / GMP with time and date.

Fig. 12 Anylytical balances

3.7 Preparation of CMC Solution and coated fabric

30 g/l solutions of CMC were prepared using six different types of CMC salts. They were labeled and stored overnight to dissolve completely in water. Distilled water was used to prepare these solutions. The solutions of these CMC salts had different viscosities depending on their molecular weights and degree of substitution.It was noted that the CMC solution prepared from salt no. 5 with the highest molecular weight was the most viscous solution of them all so as the CMC solution made from salt no. 6 which had the lowest molecular weight was also noted to have be the least viscous solution.

<u>Fixing of PES substrate with different salt solutions (30g/l)of CMC</u>

- Nonwoven PES fabric was cut into pieces of 20cm X 20cm.
- Each piece was weighed and the masses of the pieces were recorded.
- Each piece was placed on a separate container.

- CMC solution was poured over the substrate and brushed into the substrate to obtain maximum absorption of the solution by the substrate.
- The substrate samples were weighed again and weight was recorded.
- The samples were then dried in the oven at 105 °C.
- The samples were then cooled down and the weight recorded again.

Table 5 : Application of CMC Solutions onto PES substrate

Sample No.	CMC Salt	Weight of Sample(g) Before Addition of CMC	Weight of Sample(g) After Addition of CMC	Weight(g) of Sample After Drying	Weight(g) of CMC fixed onto the sample
1.	Salt No.1	3.44	204.05	9.08	5.64
2.	Salt No.2	4.12	201.43	9.13	5.01
3.	Salt No.3	3.25	196.52	8.53	5.28
4.	Salt No.4	4.52	209.51	9.72	5.20
5.	Salt No.5	3.42	202.68	9.04	5.62
6.	Salt No.6	3.78	204.96	9.27	5.49

- Each sample piece was then cut to smaller pieces for crosslinking at different temperatures.
- Each piece was cut to six pieces to be crosslinked at different temperature from 150 °C to 200 °C.

3.8 Determination of Degree of Substitution by Titration Method

- Reagents Used: Nitric Acid (65%)
  Ethanol (95%)
  Sodium Hydroxide
  Hydrochloric Acid (35%)
  Methanol
  Phenolphthalein Indicator
  Carboxymethylcellulose Salt

Absolute values of degree of substitution were determined by potentiometric titration. A portion of the carboxymethylated product was dissolved in 60mL of 95% of ethanol and stirred. 10mL of 2M nitric acid was added and the mixture was agitated for 2 min. The mixture was then heated to boiling for 5 min and agitated further for 15 min and left to settle.

After the solution had settled the supernatant liquid was filtered and discarded. The precipitate was washed with 80mL of 95% of ethanol. After washing with 80ml of 95% ethanol, a further washing with 80% ethanol which has been heated at 60°C was done to remove traces of the acid and salts. The precipitate was then washed with methanol and transferred to the beaker and heated until the alcohol was removed. The beaker with the precipitate was dried in the oven at 105 °C for 3 hours.

About 0.5g of acid carboxymethyl cellulose was weighed into a 250mL Erlenmeyer flask and 100mL of distilled water was added and stirred. 25mL of 0.5N sodium hydroxide was added and boiled for about 15 min. The heated solution was titrated with a 0.3N hydrochloric acid by using phenolphthalein as an indicator. The carboxymethyl content and the degree of substitution were calculated based on the following equations :

$$\text{Carboxymethyl Content} = [(V_0 - V_n) \times 0.058 \ \ 100] : M \quad (1)$$

$$\text{Degree of Substitution} = 162 \times \%CM : [5800 - (57 \times \%CM)] \quad (2)$$

V_0 …… mL of HCl used to titrate blank

V_n …… mL of HCl used to titrate sample

M …… mass of sample

58g/mol…. molecular weight of carboxymethylgroup

3.8.1.1 Determination of the degree of substitution

- How to prepare 2M HNO_3 :

  - 9mls of concentrated HNO_3 were used to prepare 100mL of 2M HNO_3 solution. Distilled water was used to prepare the solution.

- How to prepare 80% Ethanol from 95% Ethanol:

  - 84.21mL of 95% Ethanol were used to make 100mL of 80% Ethanol and filled to volume mark with distilled water.

- How to make 0.5N sodium hydroxide solution:

  - 0.2g of NaOH was weighed to make 100mL of a 0.5N NaOH solution.

- How to make 0.3N HCl solution:

  - 10.79ml of concentrated HCl was used to prepare 100ml of 0.3N HCl. The titration was done three times and the average was calculated.

Masses of CMC were weighed as follows :

M_1 = 0.50g V_n = 4.50ml V_0 = 8.00ml

M_2 = 0.52g V_n = 4.60ml

M_3 = 0.51g V_n = 4.70ml

1) %CM = $[(V_0 - V_n) \times 0.058 \times 100] : M$

$= [(8 - 4.50) \times 0.058 \times 100] : 58$

$= 0.35\%$

2) %CM = $[(V_0 - V_n) \times 0.058 \times 100] : M$

$= [(8 - 4.60) \times 0.058 \times 100] : 58$

$= 0.34\%$

3) %CM = $[(V_0 - V_n) \times 0.058 \times 100] : M$

$= [(8 - 4.70) \times 0.058 \times 100] : 58$

$= 0.33\%$

Table 6 : Results of %CM and DS of salt number 3

	1	2	3
%CM	0,35	0,34	0,33
DS	0,981	0,953	0,925

Average degree of substitution = 0.953

3.9 **Preparation of CMC Hydrogels using Epichlorohydrin**

(as a crosslinking agent)

Superabsorbent hydrogels were prepared successfully from carboxymethylcellulose sodium (CMC) and cellulose in the NaOH/urea aqueous system by using epichlorohydrin (ECH) as cross-linker. CMC contributed to the enhanced size of pore. Their equilibrium swelling ratio in distilled water and different physiological fluids were evaluated, indicating the maximum swelling ratio in water reached an exciting level of 1000 as the hydrogels still keeping a steady appearance. Moreover, the hydrogels exhibited smart swelling and shrinking in NaCl or CaCl2 aqueous solutionas well as their release behaviour which is very good. [19]

The cellulose-based hydrogels are promising for the applications in the biomaterials area. Superabsorbent hydrogels are three-dimensional crosslinked hydrophilic, linear or branched polymers with the ability to absorb large quantities of water. compared with general absorbing materials Because of their excellent hydrophilic properties, high swelling ratio, and biocompatibility, hydrogels have been widely used in agriculture. Biodegradable superabsorbent hydrogels have been prepared through etherifying of the cellulose with succinic anhydride, which can absorb an amount of water of about 400 times of its dry weight. [20]

Hydrogel of many synthetic and natural polymers have been produced with their end use mainly in tissue engineering, pharmaceutical, and biomedical fields. Due to their high water absorption capacity and biocompatibility they have been used in wound dressing, drug delivery, agriculture, sanitary pads as well as trans-dermal systems,dental materials, implants, injectable polymeric systems, ophthalmic applications, [21]

3.10 ICP OES analysis

The method of optical emission spectrometry with inductively coupled plasma (ICP OES) is used mainly for elemental analysis of geomaterials. The method allows determination of a wide range of major , minor and trace elements.

The ICP spectrometer by radial plasma observation allows the determination of elements in the complex (geo) matrices. The spectrometer uses the principle of emissions and subsequent detection of ionized radiation of light elements present in the sample. Ion source is argon plasma. This is the method of solutions, therefore it is necessary to convert solid samples into solution. ICP OES can be connected with the laser probe NewWave 213 nm and subsequently analyzed aerosol from a solid sample.

Figure 13: ICP OES instrument

4 Results and discussion

Objectives of the experiment

- The first objective of the experiment was to determine the crosslinking temperature influence on the % absorption capacity of the treated fabric.
- The second objective was to determine the effect of time of crosslinking of the sample on the % absorption capacity of the treated fabric.
- The third objective was to observe the solubility of CMC with time.
- The fourth objective was to observe the effect of the chemical crosslinking of CMC using epichlorohydrin.

4.1.1 Measurement of absorption and solubility

The following method was used to determine the solubility of CMC in water. Samples which had been crosslinked at different temperatures were cut into pieces and weighed. They were then dried at 105 °C for 1hour and 30 minutes using special glass bottles with lids as shown in Fig. 11. to avoid moisture from the atmosphere to be absorbed. After that were samples crosslinked in the heated box at different temperatures from 150 °C to 200 °C for a variol period of time. The weight of each dried sample, determined to ± 0.01 g, was taken as the initial dry weight of the sample (M). After drying the sample was immersed in distilled water in a crucible for 1 hour. Samples were then removed and weighed again and the second weight was recorded as (W_2). The % solubility was then calculated using the following equation.

$$\%\text{Solubility} = (M_2 - M_1) / M_2 * 100 \quad (3)$$

M_2 =Mass after polymerisation/crosslinking

M_1 =Mass after immersion in water and dryeing

$$\%\ \text{Absorption} = (M_3 - M_4) / M_4 * 100 \quad (4)$$

M_4 =Mass after drying

M_3 =Mass after immersing in water

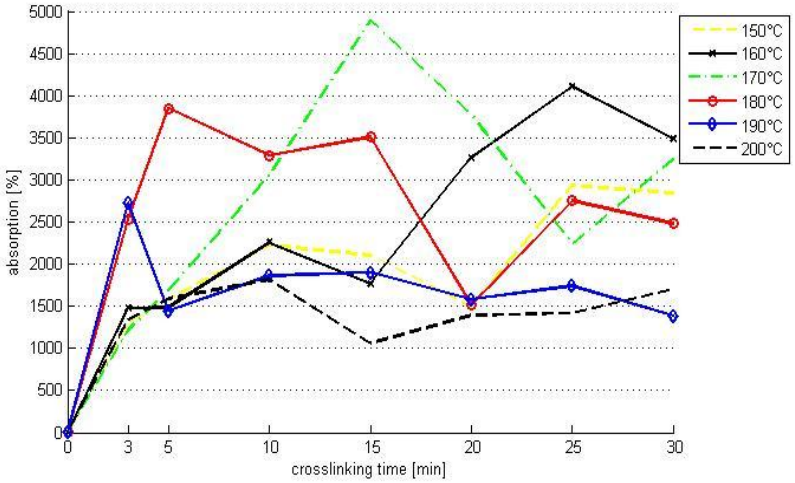
Fig. 14: Special glass bottles with lids

- Inerting Samples at different times : (samples)
  - No. 1 - crosslinked for 1 min
  - No. 2 - crosslinked for 3 min
  - No. 3 - crosslinked for 15 min
  - No. 4 - crosslinked for 10 min
  - No. 5 - crosslinked for 15 min
  - No. 6 - crosslinked for 20 min
  - No. 7 - crosslinked for 25 min
  - No. 8 - crosslinked for 30 min

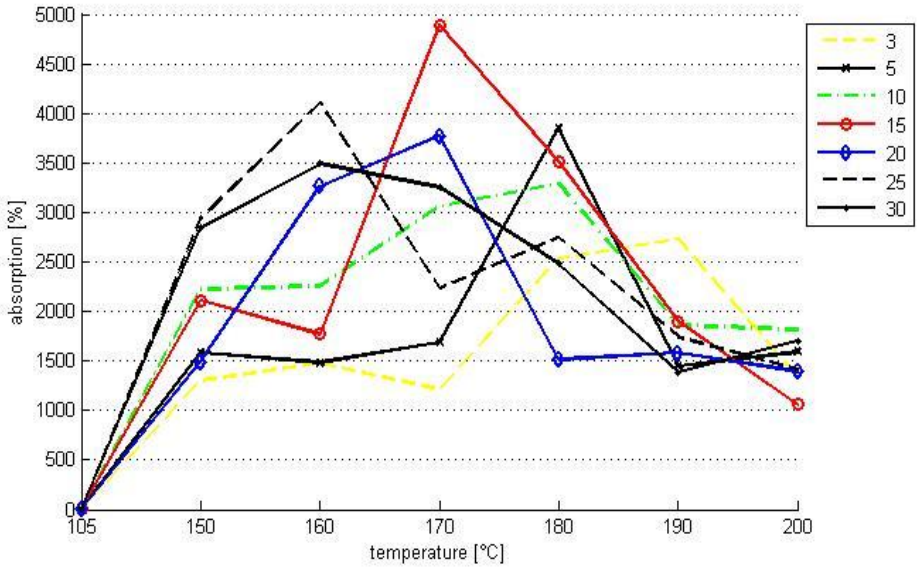
Below are the graphical presentations that show the relationship between % absorption capacity , temperature and time.

4.1.2 CMC No. 1

4.1.2.1 Absorption



Graph 1: Absorption as a function of crosslinking time – CMC salt No. 1



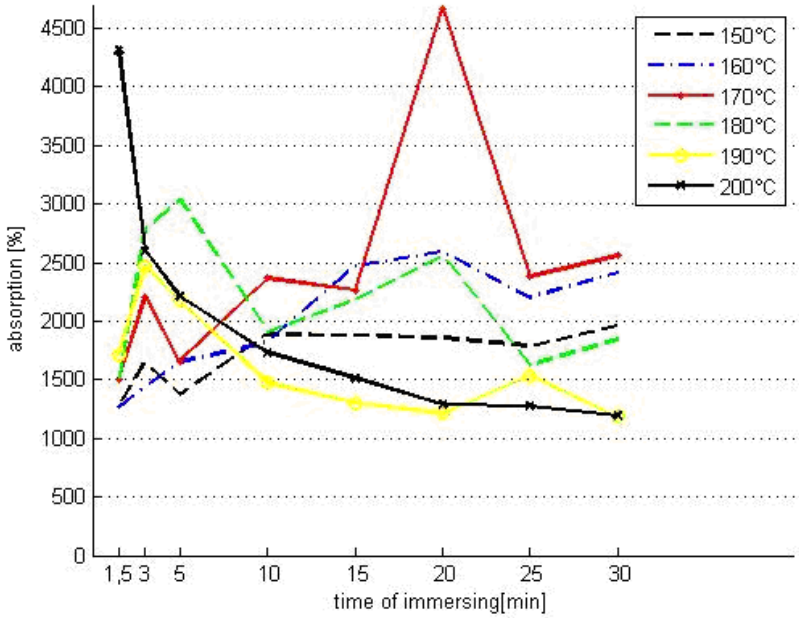
Graph 2: Absorption as a function of crosslinking temperature – CMC salt No. 1

Observation:

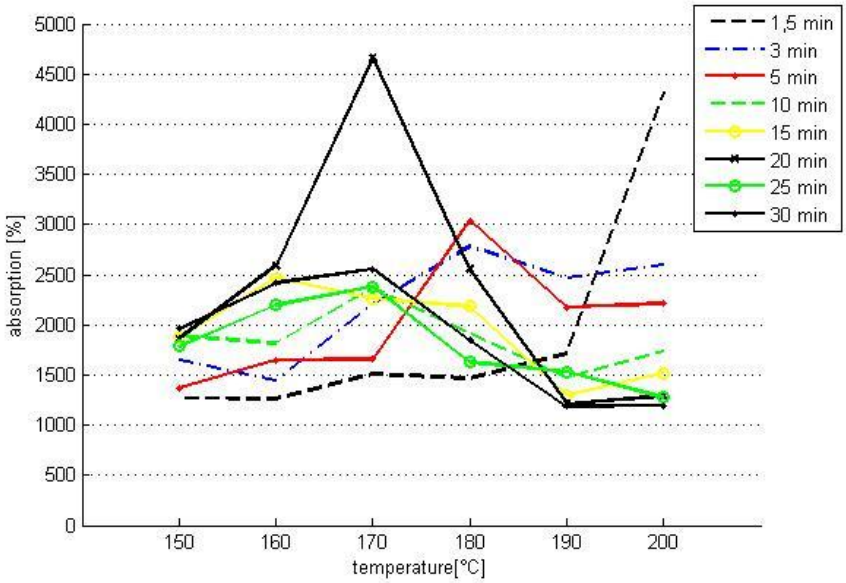
Sample crosslinked at 170 °C has the maximum absorption capacity at the time of about 15 minutes.

4.1.3 CMC No. 2

4.1.3.1 Absorption

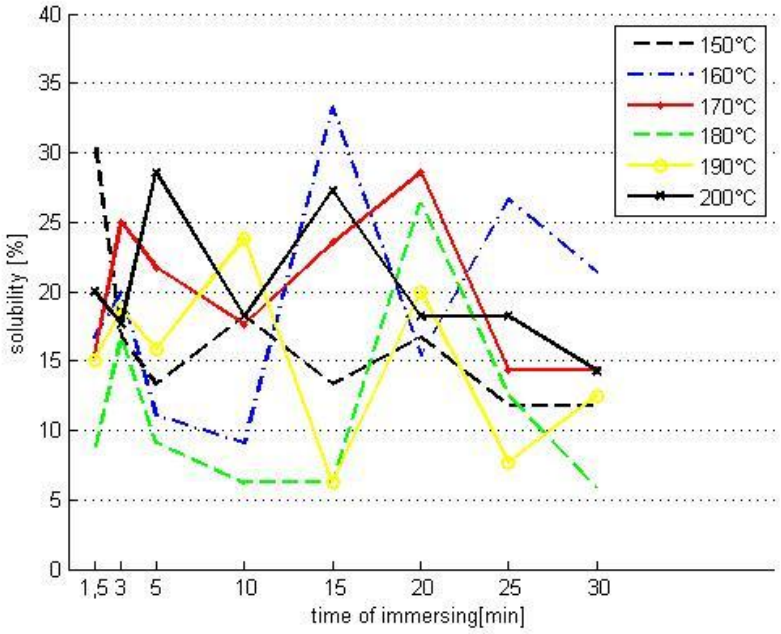


Graph 3: Absorption as a function of crosslinking time – CMC salt No. 2



Graph 4: Absorption as a function of crosslinking temperature – CMC salt No. 2

4.1.3.2 Solubility



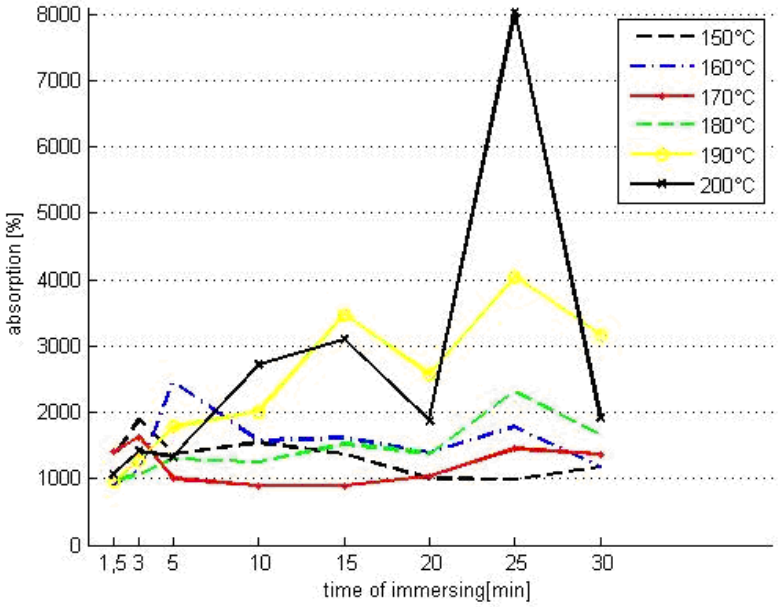
Graph 5: Solubility as a function of crosslinking time – CMC salt No. 2

Observation :

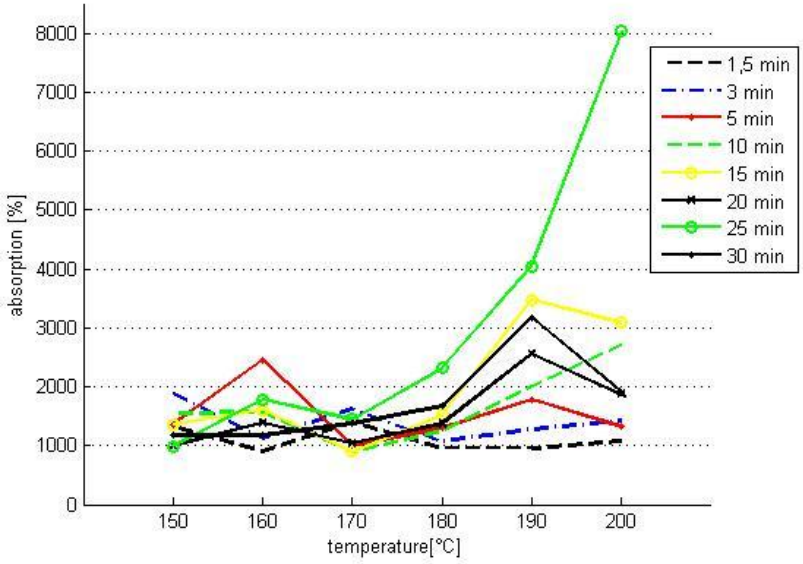
Sample crosslinked at 170 °C has the maximum absorption capacity at the time of about 20 minutes. Sample crosslinked at 160 °C has maximum solubility at the time around 15 minutes of immersing in water.

4.1.4 CMC No. 3

4.1.4.1 Absorption

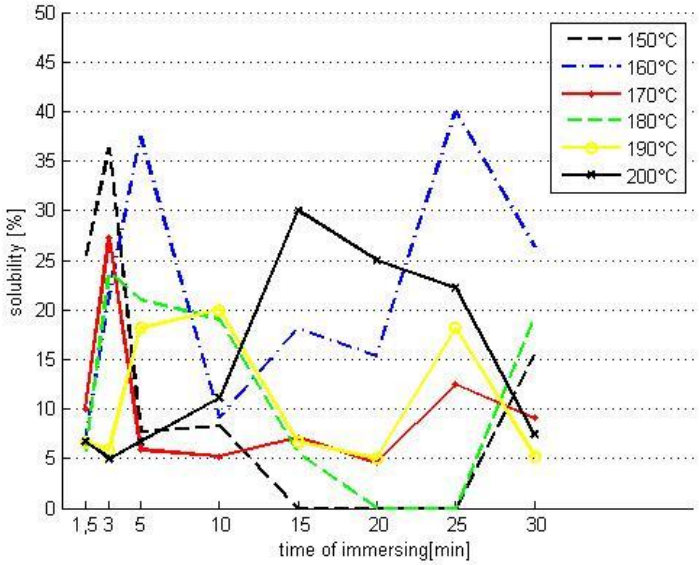


Graph 6: Absorption as a function of crosslinking time – CMC salt No. 3



Graph 7: Absorption as a function of crosslinking temperature – CMC salt No. 3

4.1.4.2 Solubility



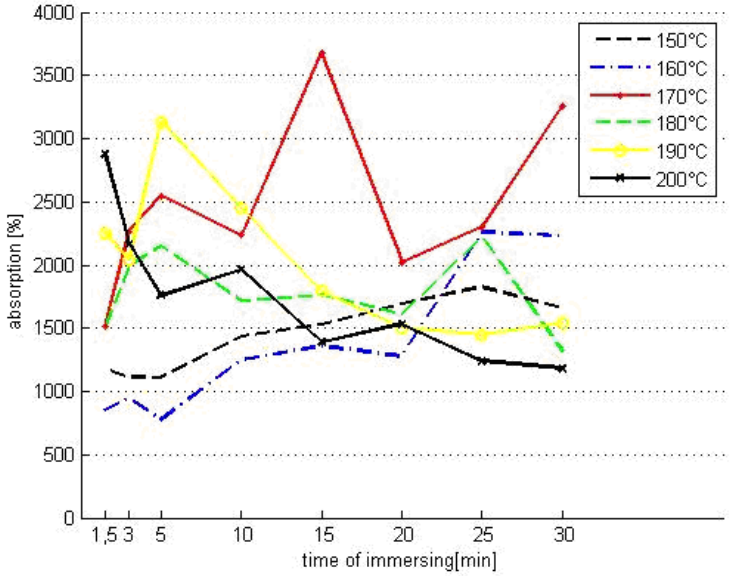
Graph 8: Solubility as a function of crosslinking time – CMC salt No. 3

Observation :

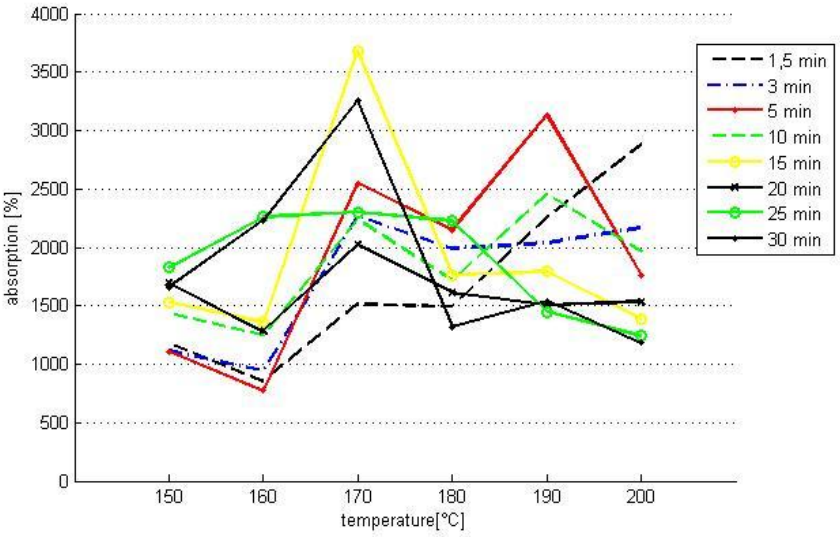
Sample crosslinked at 200 °C has the maximum absorption capacity at the time of about 25 minutes. Sample crosslinked at 160 °C has maximum solubility at the time around 25 minutes of immersing in water.

4.1.5 CMC No. 4

4.1.5.1 Absorption

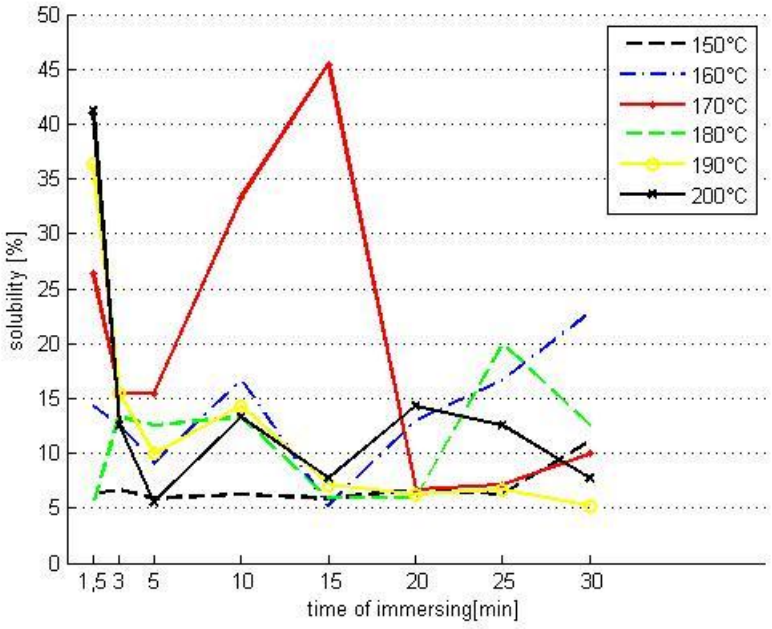


Graph 9: Absorption as a function of crosslinking time – CMC salt No. 4



Graph 10: Absorption as a function of crosslinking temperature – CMC salt No.4

4.1.5.2 Solubility



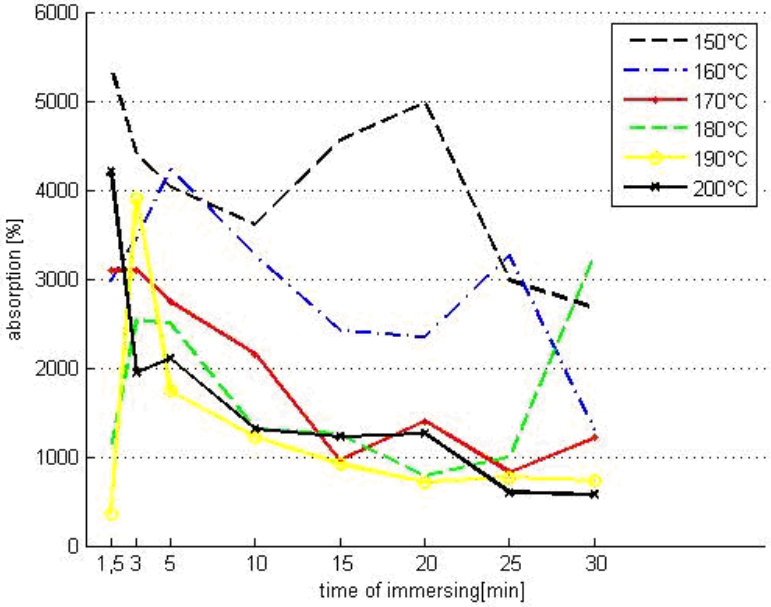
Graph 11: Solubility as a function of crosslinking time – CMC salt No. 4

Observation :

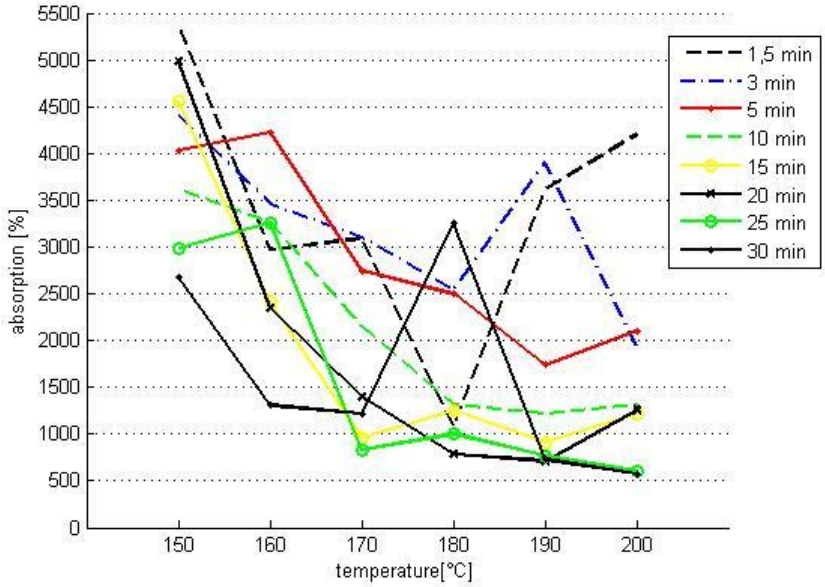
Sample crosslinked at 170 °C has the maximum absorption capacity at the time of about 15 minutes. Sample crosslinked at 170 °C has maximum solubility at the time around 15 minutes of immersing in water.

4.1.6 CMC No. 5

4.1.6.1 Absorption

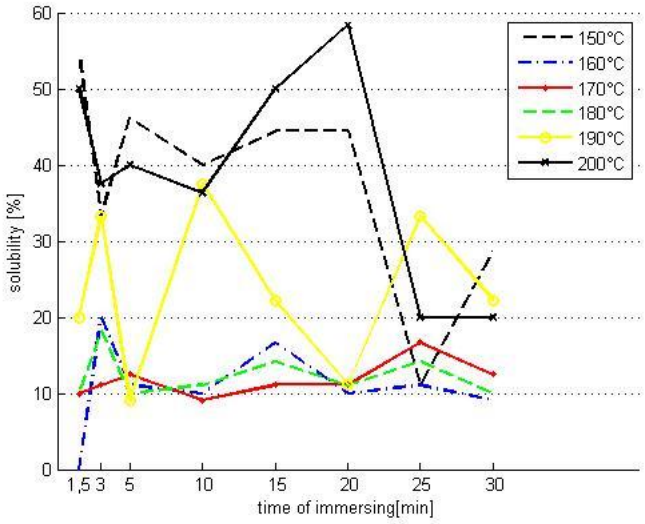


Graph 12: Absorption as a function of crosslinking time – CMC salt No. 5



Graph 13: Absorption as a function of crosslinking temperature – CMC salt No. 5

4.1.6.2 Solubility



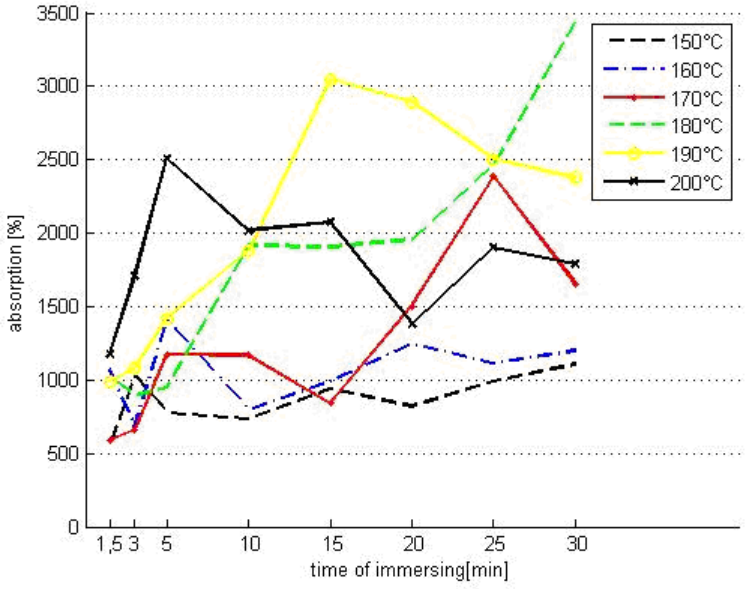
Graph 14: Solubility as a function of crosslinking time – CMC salt No. 5

Observation :

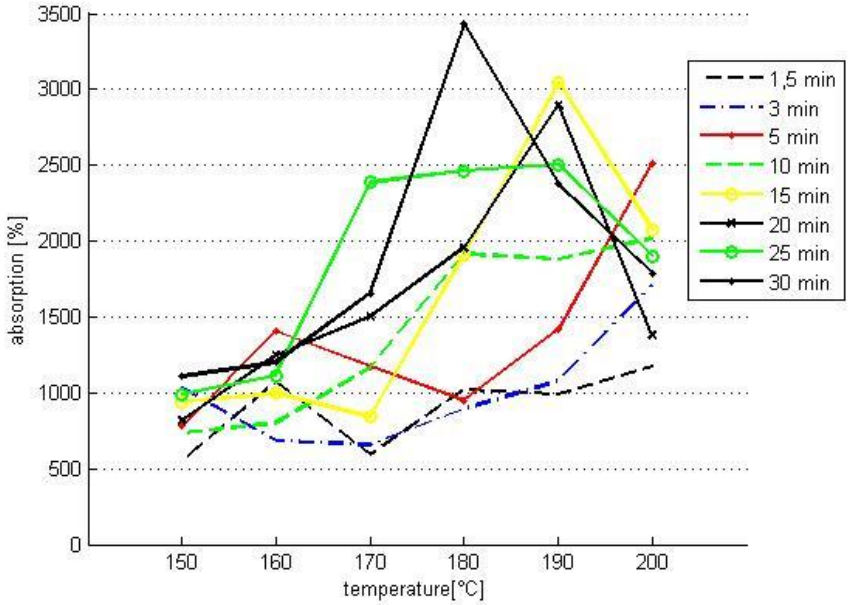
Sample crosslinked at 150 °C has the maximum absorption capacity at the time of about 20 minutes. Sample crosslinked at 200 °C has maximum solubility at the time around 20 minutes of immersing in water.

4.1.7 CMC No. 6

4.1.7.1 Absorption

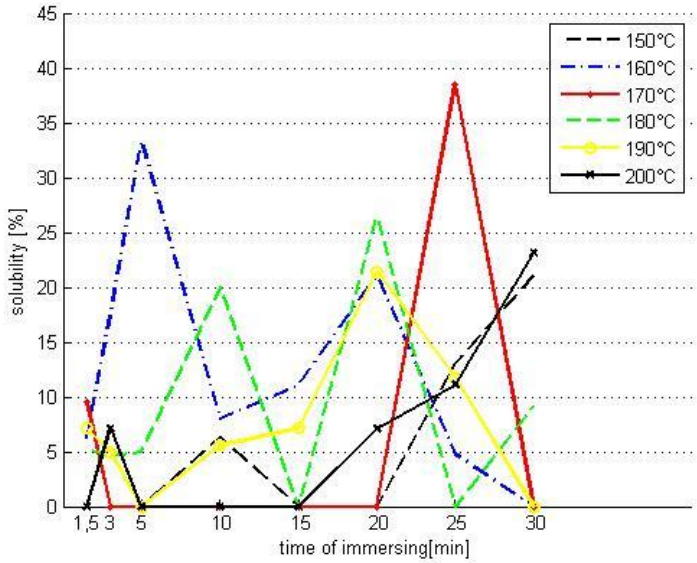


Graph 15: Absorption as a function of crosslinking time – CMC salt No. 6



Graph 16: Absorption as a function of crosslinking temperature – CMC salt No. 6

4.1.7.2 Solubility



Graph 17: Solubility as a function of crosslinking time – CMC salt No. 6

Observation :

Sample crosslinked at 180 °C has the maximum absorption capacity at the time of about 15 minutes. Sample crosslinked at 170 °C has maximum solubility at the time around 25 minutes of immersing in water.

From the graphs of temperature dependency which we obtained, we noticed that all the salts that were used had the best crosslinking effect which was clearly displayed by the maximum % absorption capacity between 170 °C and 180 °C.

Therefore we can conclude that the best crosslinking temperature of CMC onto the PES fabric is between 170 °C and 180 °C.

From the graphs of time dependency which we obtained, we noticed that all the salts that were used had the best crosslinking effect which was clearly displayed by the maximum % absorption capacity between 15minutes to 25 minutes. Therefore we can conlude that the best crosslinking time of CMC onto the PES fabric is between 15 minutes and 25 minutes.

From the graphs of time dependency which we obtained we noticed that all the salts that were used had the maximum % solubility which was clearly displayed by the time around 10minutes to 20 minutes.

4.2 Absorption Capacity of the Untreated Fabric

A sample of the untreated nonwoven polyester was cut into small pieces which were weighed before immersing in water. The pieces were then immersed in water for 1 hour and weighed again. The amount of water which samples could retain was less than the amount of water that the treated sample can hold.

Table 7 : Weight of samples before and after immersing

Sample No.	Weight (g) Before(W_1)	Weight (g) after(W_2)
1.	0.18	5.85
2.	0.17	5.23
3.	0.15	7.03

The average % absorption was calculated and found to be 3571%

Using the following equation :

$$\% \text{ Absorption} = W_2 - W_1 : W_1 \times 100 \quad (5)$$

4.3 Crosslinking of CMC with Epichlorohydrin

4.3.1 Dried Normally on the petri dish

Samples of CMC crosslinked with epichlorohydrin were cut into smaller pieces and dried at 40 °C. The weight of each dried sample, determined to ±0.01 g, was taken as the initial dry weight (W_0) of the sample. Each sample was immersed in distilled water in a crucible as shown in Appendix part for different times starting from half a minute to 1 hour and thereafter weighed to observe the amount of water absorbed. During the dissolution process, sample pieces were removed by use of a twizzer and dried at 105 °C to determine the final dry weight (W_1).

Table 8 : Weight of samples before and after immersing and drying

CMC Sample No.	Time(min)	Weight(g) Before *immersing*	Weight(g) After *immersing*	Weight(g) after drying the Sample(W_1)
1.	0.5	0.16	0.52	0.16
2.	1	0.28	0.70	0.26
3.	3	0.23	0.85	0.23
4.	5	0.14	0.78	0.12
5.	7	0.21	1.62	0.20
6.	10	0.19	1.06	0.11
7.	15	0.17	1.97	0.10
8.	20	0.12	0.80	0.04
9.	25	0.14	1.00	0.06
10.	30	0.17	2.05	0.07
11.	60	0.13	0.38	0.02

4.3.2 Sample dried between the two plates

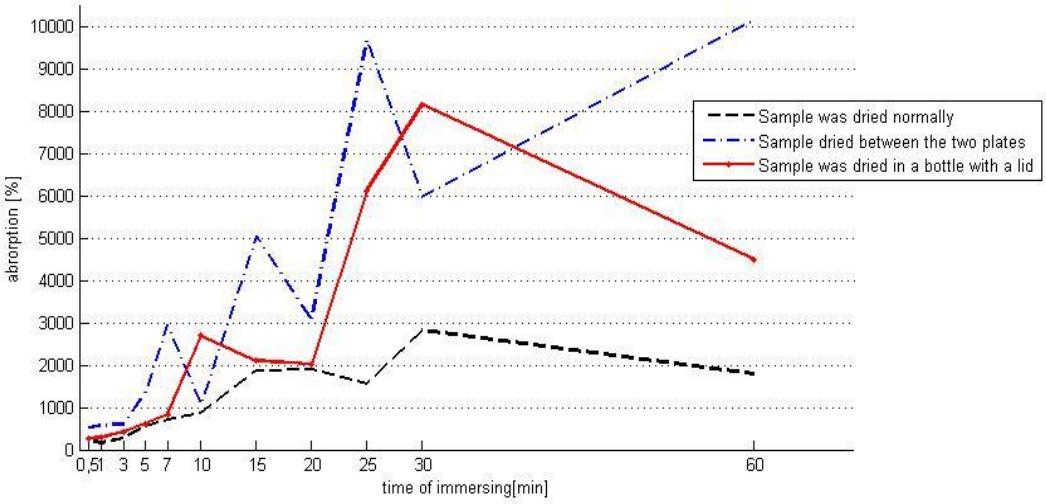
Table 9 : Weight of samples before and after immersing and drying

CMC Sample No.	Time(min)	Weight(g) Before *immersing*	Weight(g) After *immersing*	Weight(g) after drying the Sample(W_1)
1.	0.5	0.05	0.31	0.05
2.	1	0.04	0.20	0.03
3.	3	0.04	0.28	0.04
4.	5	0.10	1.16	0.08
5.	7	0.03	0.61	0.02
6.	10	0.13	1.29	0.11
7.	15	0.12	1.03	0.02
8.	20	0.08	2.22	0.07
9.	25	0.05	1.96	0.02
10.	30	0.08	2.44	0.04
11.	60	0.14	5.15	0.05

4.3.3 Sample dried in the bottle with the lid on

Table 10: Weight of samples before and after immersing and drying

CMC Sample No.	Time(min)	Weight(g) Before *immersing*	Weight(g) After *immersing*	Weight(g) after drying the Sample(W_1)
1.	0.5	0.11	0.41	0.11
2.	1	0.07	0.27	0.07
3.	3	0.17	0.86	0.16
4.	5	0.14	1.00	0.14
5.	7	0.17	1.22	0.13
6.	10	0.16	3.09	0.11
7.	15	0.16	1.99	0.09
8.	20	0.12	1.70	0.08
9.	25	0.16	4.36	0.07
10.	30	0.09	3.31	0.04
11.	60	0.14	1.84	0.04



Graph 18: Comparing absorption of 3 samples Crosslinked with Epichlorohydrin

Observation :

All three samples that were used which had been crosslinked with Epichlorohydrin show maximum absorption capacity between 25 to 30 minutes time of immersing in water.

4.4 Metal ions sorption

Samples were prepared by the same way which is described in chapter 4.1.1. Dry crosslinked samples were placed into the flasks with volume of 1litre which can be closed. Each flask obtained 1 liter of distilled water and calculated amount of metal ions salt. Amount of metal ions salt was calculated from molar weight of each salt. It was carried out a measurement of pH of each solution by pH meter. Also was made a ICP OES anylysis. Results of both measurements are shown in chapter 4.4.1 and 4.4.2.

Table 11 : Amount of salts in solutions

Metal ions salts	Molar weight [g/mol]	Amount of metal ions salt in solution [g/l]
Sn ($SnCl_2$)	266	0.18104
Cu ($CuSO_4$)	250	0.17015
Pb ($PbC_4H_6O_43H_2O$)	189	0.12863

- Amount of metal ions salt in solution = molar weight of metal ions salt . N metal [g/l]

m CMC [g]	N COOH	N metal
0.1	0.00034	0.000681

4.4.1 Samples coated with CMC (salt number 3) using foulard

Table. 12 : Samples coated with CMC using Foulard crosslinked at 160 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.15	0.14	0.2534	5.55	0.1384
Cu	0.14	0.13	0.2212	5.01	0.0614
Sn	0.15	0.15	0.1930	2.72	0.0007

Table 13 : Samples coated with CMC using Foulard crosslinked at 170 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.14	0.15	0.2712	5.49	0.1406
Cu	0.16	0.12	0.205	5.01	0.0574
Sn	0.13	0.13	0.1672	2.78	0.0004

Table 14 : Samples coated with CMC using Foulard crosslinked at 180 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.14	0.14	0.2535	5.52	0.1291
Cu	0.14	0.14	0.2382	5.41	0.0717
Sn	0.15	0.14	0.1809	2.71	0.0007

Table 15 : Samples coated with CMC using Foulard crosslinked at 190 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.12	0.10	0.1811	5.51	0.0697
Cu	0.21	0.16	0.2722	5.92	0.0749
Sn	0.11	0.10	0.1286	2,87	0.0010

Table 16 : Samples coated with CMC using Foulard crosslinked at 200 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.16	0.15	0.2712	5.25	0.1196
Cu	0.10	0.09	0.1532	5.53	0.0442
Sn	0.10	0.09	0.1158	2.89	0.0007

Table 17 : Samples coated with CMC using Foulard crosslinked at 210 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.19	0.17	0.3077	5.41	0.1290
Cu	0.13	0.12	0.2042	5.21	0.0579
Sn	0.19	0.16	0.2058	2.83	0.0002

4.4.2 Samples coated with CMC (salt number 3) not using foulard

Table 18 : Samples coated with CMC without using Foulard crosslinked at 160 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.16	0.15	0.2717	5.60	0.1146
Cu	0.16	0.16	0.2722	5.03	0.0690
Sn	0.16	0.16	0.2058	2.68	0.0004

Table 19: Samples coated with CMC without using Foulard crosslinked at 170 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.20	0.15	0.716	5.59	0.1380
Cu	0.22	0.16	0.2722	5.07	0.0761
Sn	0.21	0.16	0.2058	2.82	0.0004

Table 20 : Samples coated with CMC without using Foulard crosslinked at 180 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.19	0.17	0.3078	5.98	0.1385
Cu	0.18	0.15	0.2552	5.13	0.0723
Sn	0.18	0.14	0.1801	2.76	0.0003

Table 21: Samples coated with CMC without using Foulard crosslinked at 190 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.21	0.18	0.3258	5.61	0.1435
Cu	0.19	0.18	0.3063	5.92	0.0968
Sn	0.21	0.18	0.2316	2.69	0.0007

Table 22 : Samples coated with CMC without using Foulard crosslinked at 200 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.20	0.19	0.3440	5.58	0.1767
Cu	0.25	0.22	0.3743	5.11	0.1004
Sn	0.25	0.21	0.2701	2.7	0.0007

Table 23 : Samples coated with CMC without using Foulard crosslinked at 210 °C

Metal ions	Weight of CMC sample (g) before	Weight of CMC sample(g) after	Weight of metal salt (g)	pH of solution	Weight of metal Salt after the reaction(g)
Pb	0.23	0.20	0.3621	5.63	0.1682
Cu	0.23	0.21	0.3573	5.03	0.0945
Sn	0.21	0.20	0.2573	2.79	0.0003

For a possibility of comparison it was also made a series of solutions with 100%, 50% and 25% amount of metal ions salts. The measurement of pH was also made.

Table 24 : pH of solutions with different amount of metal ions salts

Metal ions	100% of amount	50% of amount	25% of amount
Pb	5.06	5.44	5.54
Cu	4.89	5.00	5.19
Sn	2.89	3.07	3.39

As you can see in tables 11-23 samples of CMC are really working as a metal ions remover. The pH of solutions with Pb and Cu with CMC is very often less than at solutions with 25% of amount of metal ions salts.

5 Conclusion

Six different types of CMC salts were used in this research to have comparable results. These salts had different molecular weights and different degree of substitution as we have mentioned in the introduction that the properties of CMC depend upon three factors ie. molecular weight of the polymer, average number of carboxyl content per anhydroglucose unit, and the distribution of carboxyl substituents along the polymer chains. The most important properties of CMC are viscosity building and flocculation.

Solutions of these CMC salts had different viscosities depending on their molecular weights and degree of substitution.It was noted that the CMC solution prepared from salt no. 5 with the highest molecular weight was the most viscous solution of them all so as the CMC solution made from salt no. 6 which had the lowest molecular weight was also noted to have be the least viscous solution.

Superabsorbent hydrogels were fabricated successfully from CMC in NaOH aqueous solution by cross-linking with ECH. These hydrogels were found to absorb a significant amount of water as displayed by the % absorption graphs of CMC samples crosslinked with Epichlorohydrin as a function of time. The hydrogels absorbed nearly three times the amount of water compared to the fabric samples that were crosslinked with CMC thermally. They were also found to solubilize quicky which makes them more biodegradably.

From the results of metal analysis which we obtained, as shown above it confirms that carboxymethyl salts also possesses good complexation ability for some metal ions and can be chosen as the macromolecular complexing agent for the selective removal and rejection of copper, lead and tin and other trace metals.

Since we managed to identify the best temperature and time for crosslinking of CMC as stated above, the hydrogels will find more use in different sectors because of their ability to absorb more water and solubilize quickly, which means that they are more biodegradably.

6. References

[1] Jun-Feng, S., Zhen, H., et al.: *Carbohydrate Polymers,* Institute of Materials Science & Chemical Engineering China, Volume 79,Issue1,pg 145-153, 5 January 2010.

[2] Adel, M., Abou-Youssef, A., et al.: *Carboxymethylated Cellulose Hydrogel; Sorption Behavior and Characterization,* Cellulose and Paper Department, National Research Centre, Cairo, Egypt, Volume 8, Issue 8,2010

[3] Crammer, J., Wyandotte, E.,et al.: *Method of sizing textiles and sizing composition therefor.* 1956 No.580,622 (2811462).April 25.

[4] Barba, C., Montane, D.,.et al.: *Chemistry of Textile Finishing,* Rovira I Virgili University, Department of Chemical Engineering, 25 March 2002.

[5] Benchabade, A.: *Rheological Properties of carboxymethyl solutions,* Colloid and Polymer Science , Chemistry and Material Science Institute, Vol.286, Isssue No. 10.2008, pages 1173-1180.

[6] Sierks, M., Reily, P.: *Application of Crosslinked CMC Degradation by B-Glucosidase and Vaginal Microbes to Toxic Shock Syndrome.* Department of Chemical Engineering Lowa State University, American society for Microbiology, Vol. 50, No.3, p634-637, September 1985.

[7] Biswal, D., Singh, R.:*Carbohydrate,* Material Science Indiana Institute, Vol.57,2004 Pages 379-387.

[8] Hoelfer, A.: S*odium Carboxymethylcellulose Uses and Applications,* Hercules Incorporated Wilmington, Delaware 19808 , 01 March 2012.

[9] Hollabaugh, C., Leland, H.,et al.: *Carboxymethycellulose*, Hercules Powder company,943,October 1943.

[10] Tomasino, C.: *Chemistry and Technology of Fabric Preparation & Finishing,* Chemistry & Science College of Textiles North Carolina State University Raleigh Department of Textile Engineering,18 June 2000.

[11] Huarong, N.,Mingzhu, L.,et al.:*Carbohydrate Polymers*,Sciencedirect, Vol.58,2004,Pages 185-189

[12] Chang, Ch., Duan, B., et al.: *Superabsorbent hydrogels based on cellulose for smart swelling and controllable delivery*,European Polymer Journal, Vol 46,Issue 1 January 2010, Pages 92 – 10

[13] Butun,S., GulInce, F.,et al.:*Carbohydrate polymers*, Science direct, Vol.86,2011,Pages 636-643

[14] Clinton, M.,Granb, E.: *Crosslinking of CMC.*Rockbestos-Suprenant Cable Corp.2003.

[15] Hegde, R., Dahiya, A.,et al.:*Polyester fibers*, April 2004

[16] Wiley, J.: *Polymer Grafting and Crosslinking.*Public Health of Belgrade, Bul.Despota Stefana 54a and Paramita RayCopyright.2009.

[17] Trivunaci, K.,Sekulic, Z.,et al.: *Removal of Metals.* Faculty of Technology and Metallurgy, Department ofAnalytical Chemistry and Quality Control, Karnegijeva 4, 11120 Belgrade, Serbia and 2Institute, of Public Health of Belgrade, Bul. Despota Stefana 54a.

[18] Guo, Z., Saunders, Z.,et al.*:Properties of Lead*. Surrey Technology Centre, The Surrey Research Park, Guildford, Surrey, U.K.Vol.37 No.1,2008.Pages 23-31.

[19] Sharafat, S., Ghoniem, N.: *Summary of Thermo-Physical Properties of Sn.*

[20] Gulrez, S., Al-Assaf, S.,et al.: *Hydrogels: Methods of Preparation*

[21] Syed K. H. Gulrez, Saphwan Al-Assaf.et al. *Hydrogels: Methods of Preparation*

7. List of figures

8. List of tables

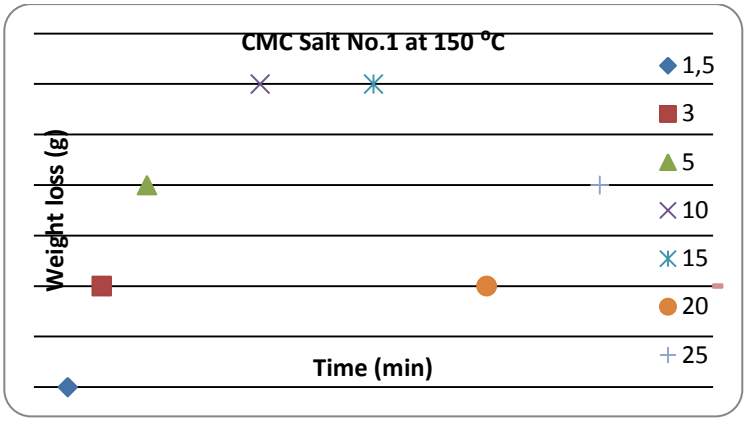
9. List of graphs

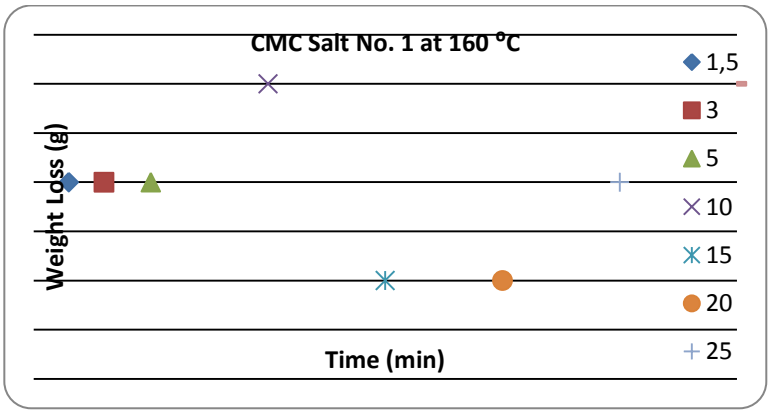
10. Appendices

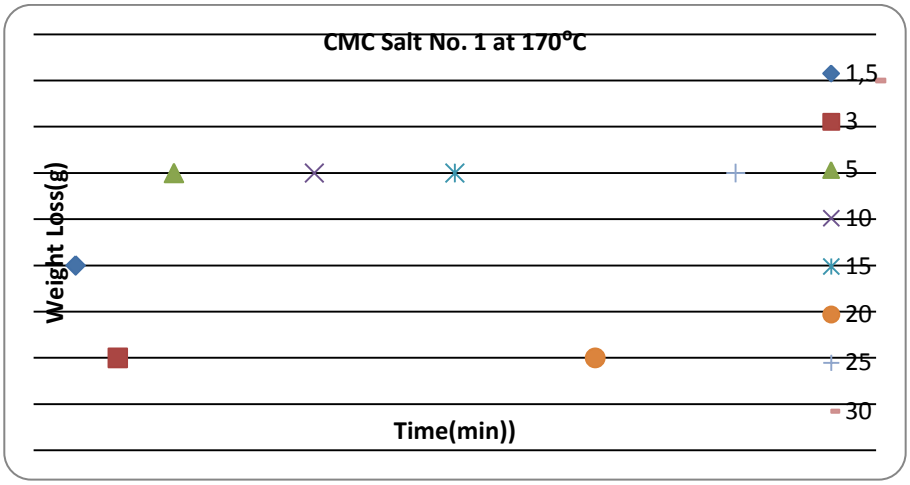
Appendix 1

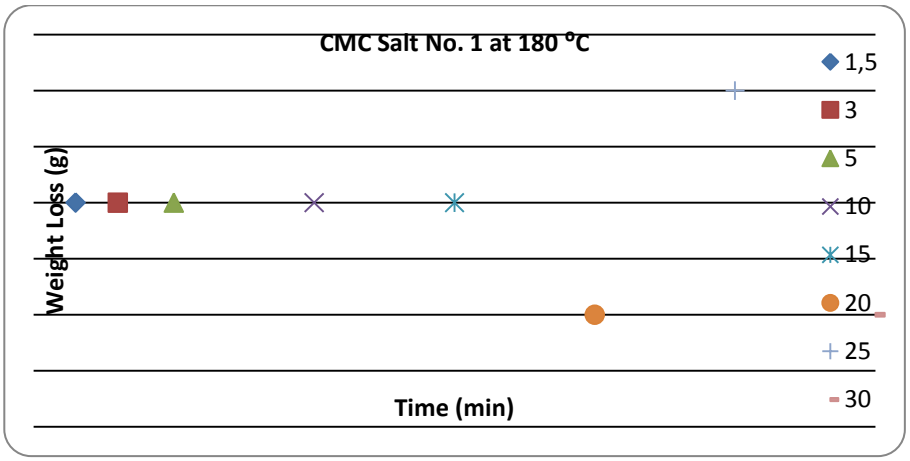
The following graphs show the advantage of using special glass bottles with lids as is shown in charter 3. during the further heating of samples for 1 hour and 30 minutes. The excess weight loss of moisture in the samples was observed for all samples crosslinked at different temperatures.

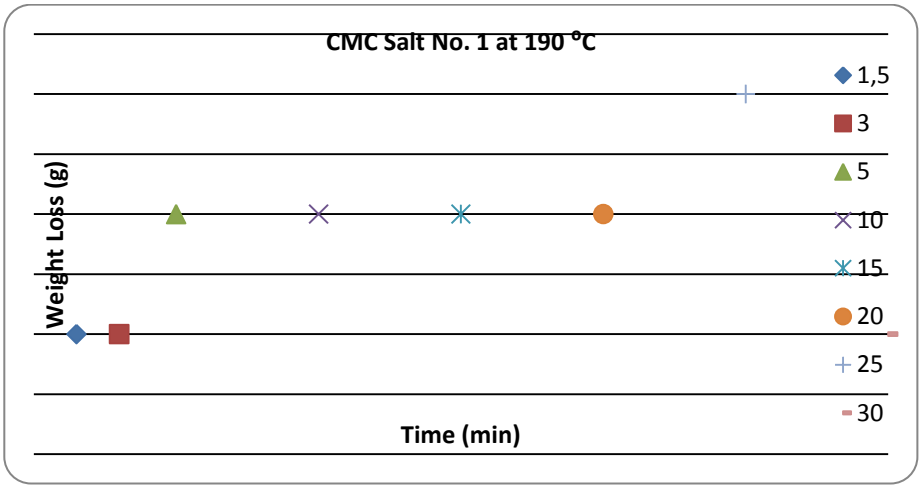
Graph of Weight Loss vs Time of samples that were crosslinked at different temperatures using salt No. 1:

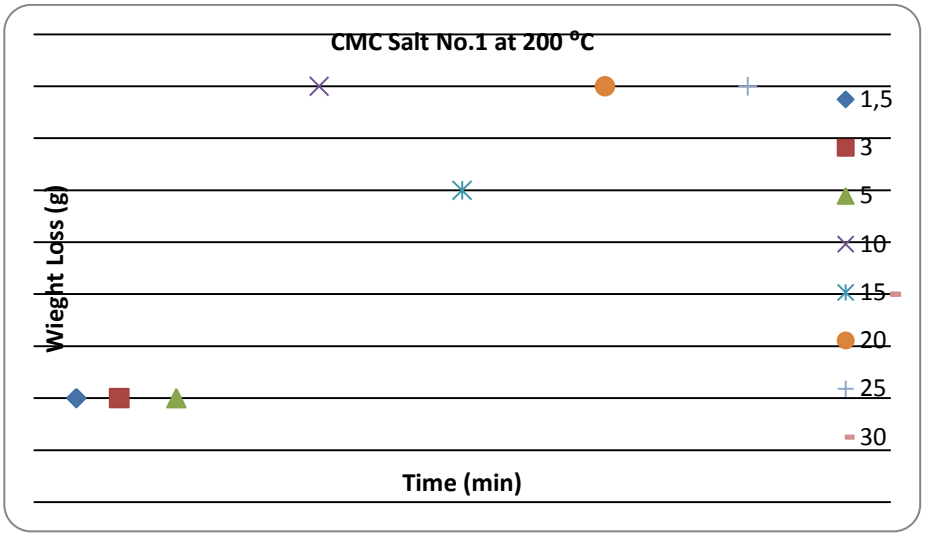




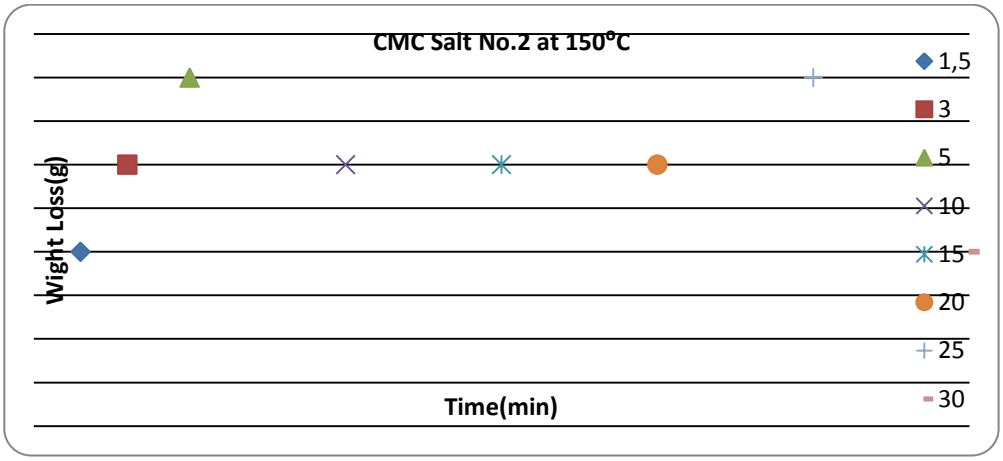

CMC Salt No. 1 at 170°C
Weight Loss(g)
Time(min))
1,5
3
5
10
15
20
25
30

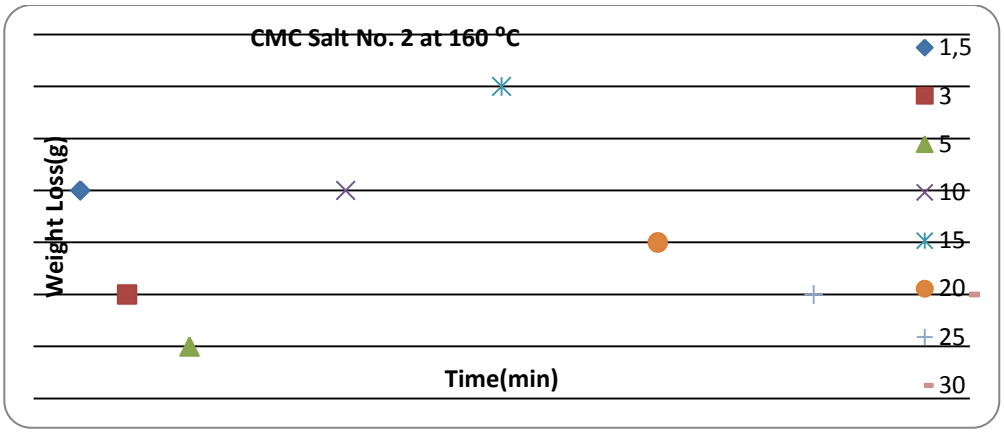

CMC Salt No. 1 at 180 °C
Weight Loss (g)
Time (min)
1,5
3
5
10
15
20
25
30

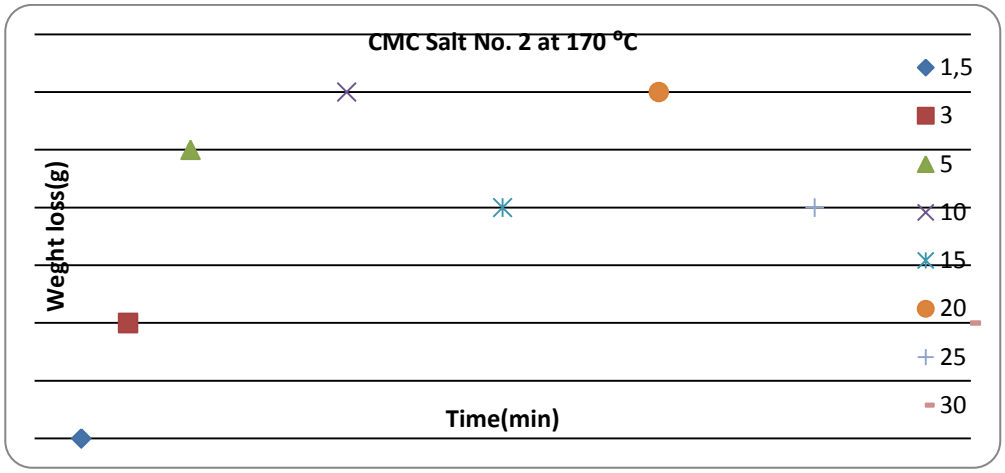

CMC Salt No. 1 at 190 °C
Weight Loss (g)
Time (min)
1,5
3
5
10
15
20
25
30

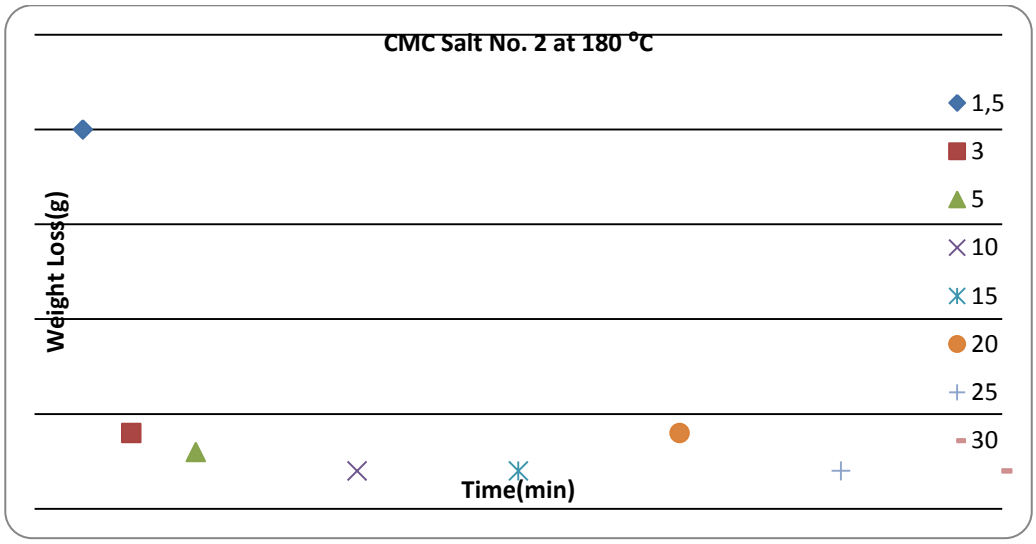


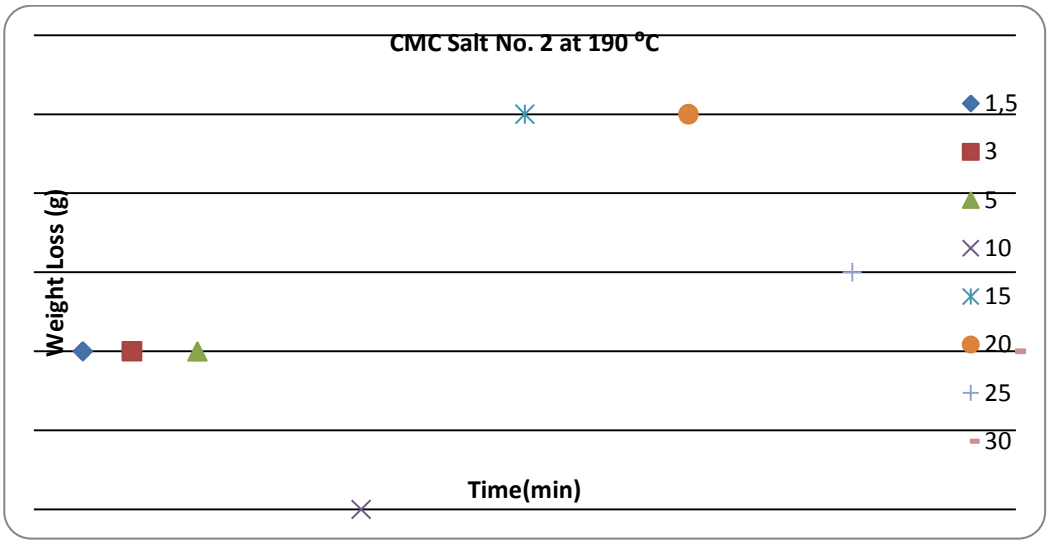
Graph of Weight Loss vs Time of samples that were crosslinked at different temperatures using salt No. 2:

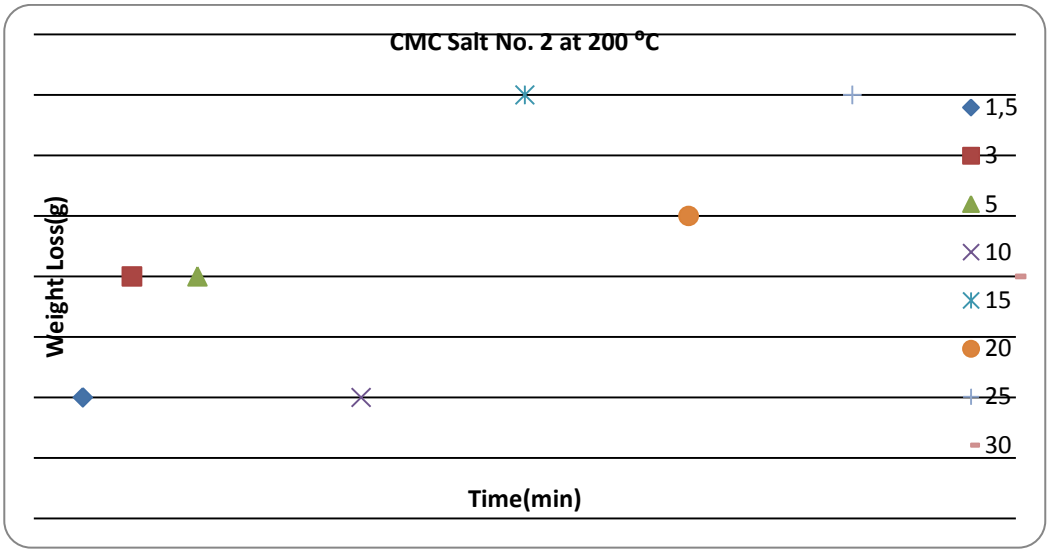




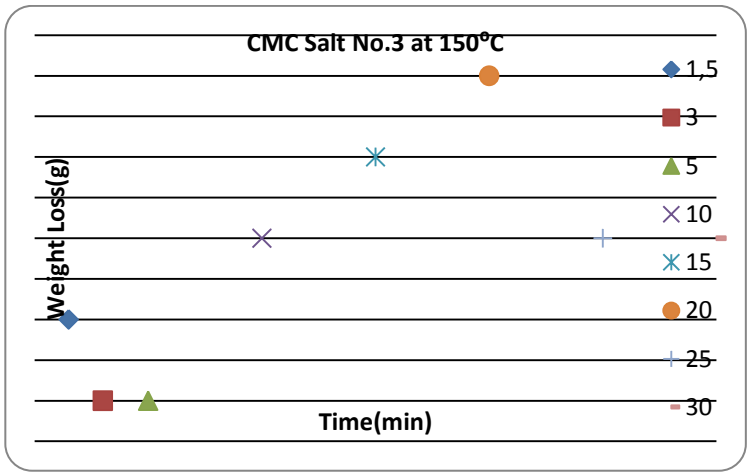

CMC Salt No. 2 at 170 °C
Weight loss(g)
Time(min)
1,5
3
5
10
15
20
25
30

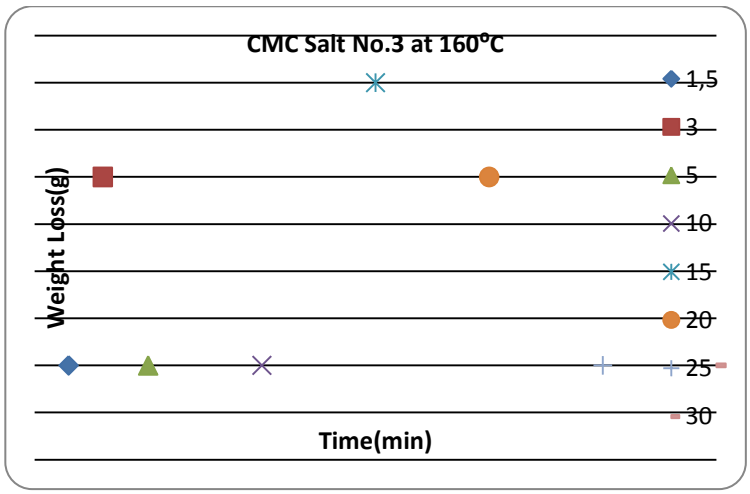

CMC Salt No. 2 at 180 °C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30

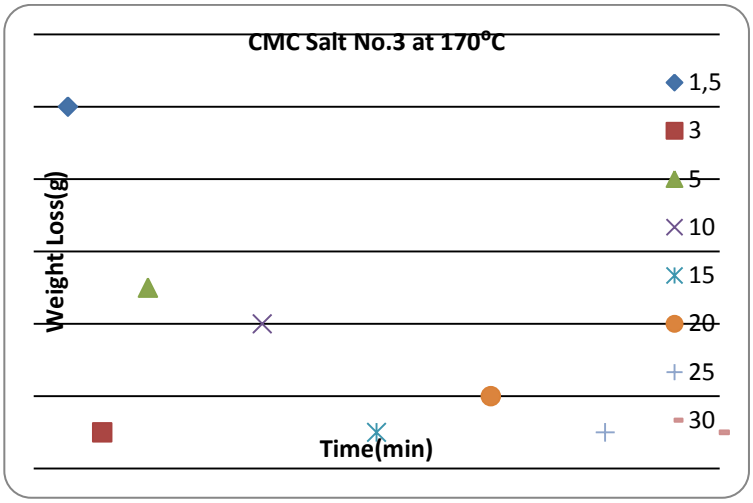

CMC Salt No. 2 at 190 °C
Weight Loss (g)
Time(min)
1,5
3
5
10
15
20
25
30

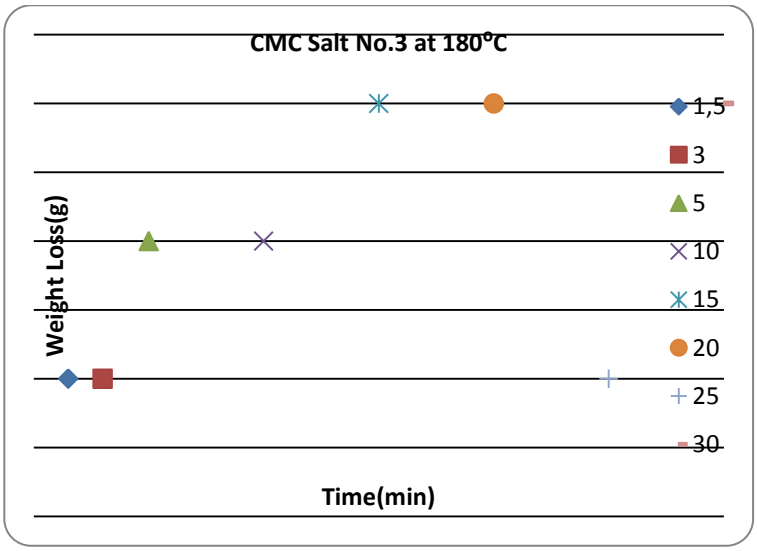


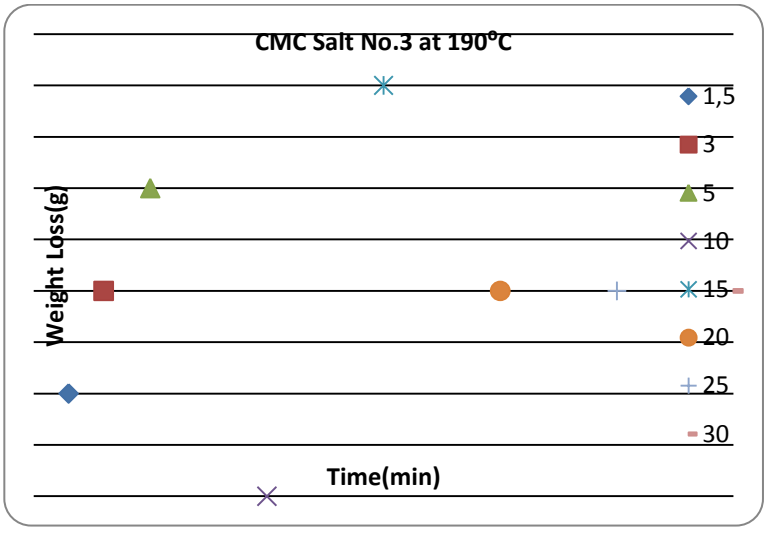
Graph of Weight Loss vs Time of samples that were crosslinked at different temperatures using salt No. 3:

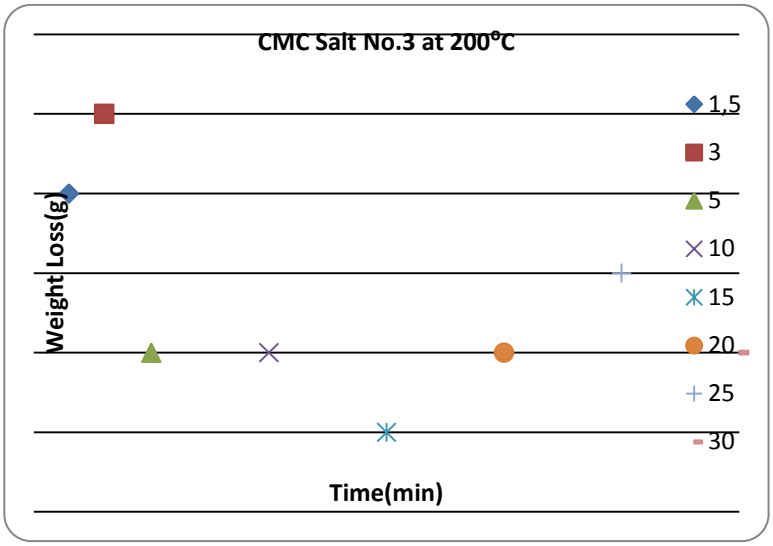




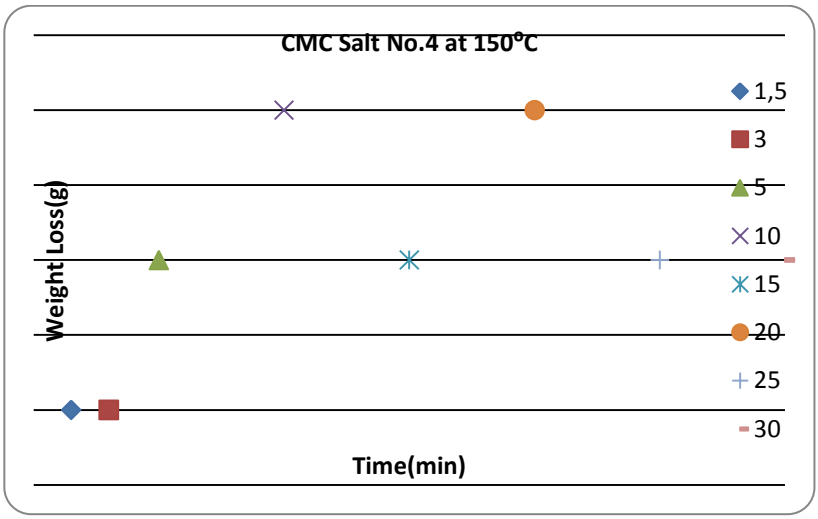

CMC Salt No.3 at 170°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30

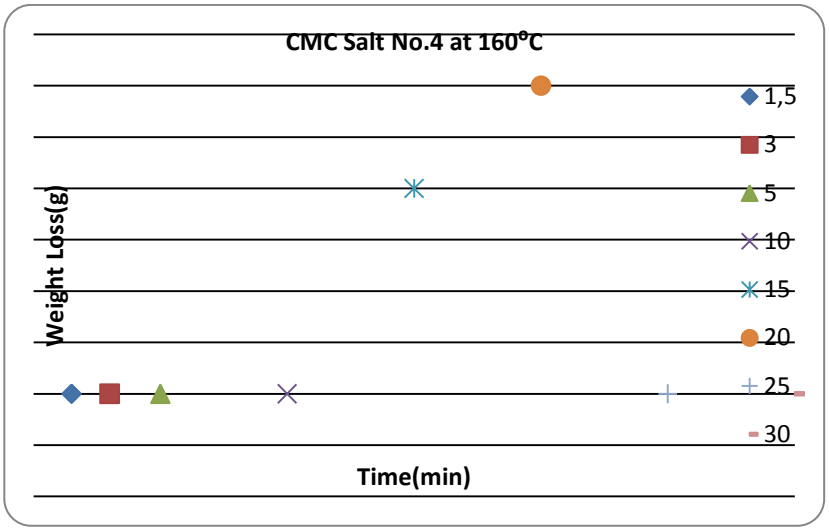

CMC Salt No.3 at 180°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30

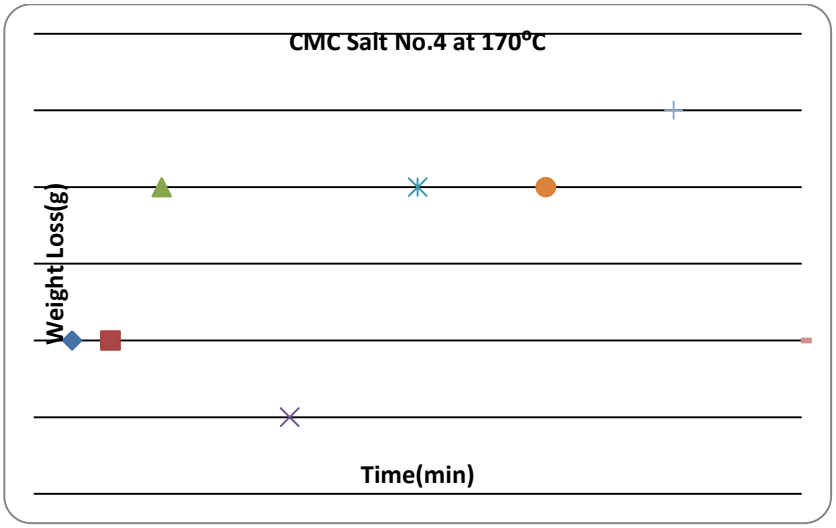

CMC Salt No.3 at 190°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30

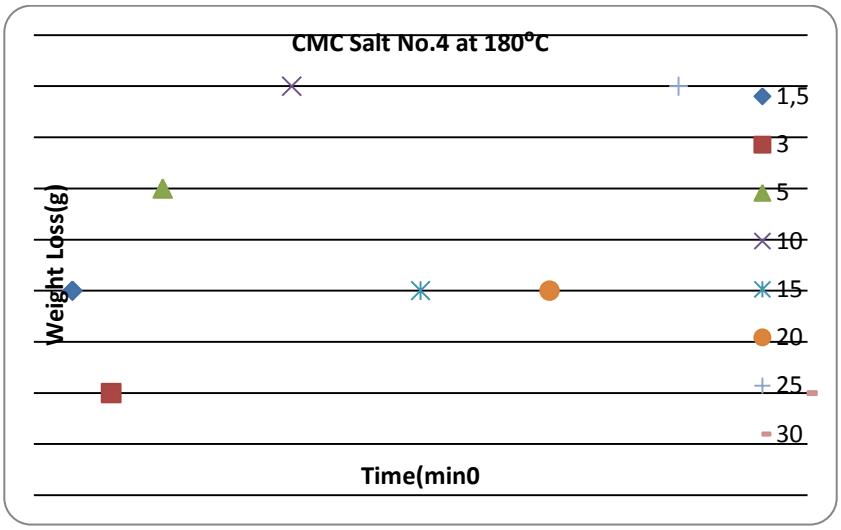


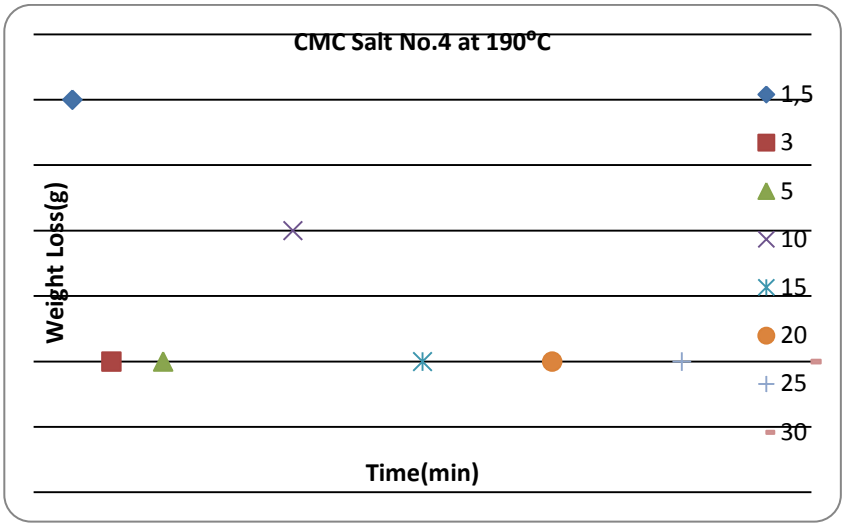
Graph of Weight Loss vs Time of samples that were crosslinked at different temperatures using salt No. 4:

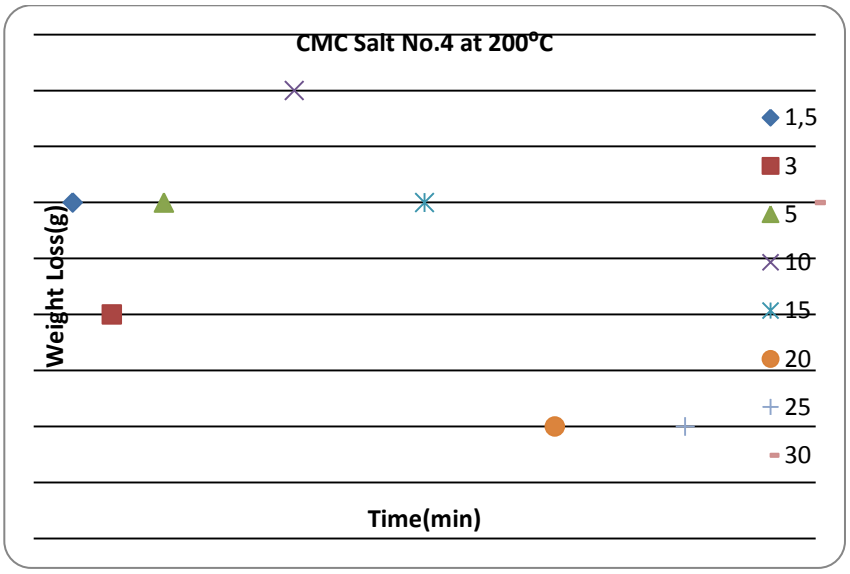



CMC Salt No.4 at 160°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30

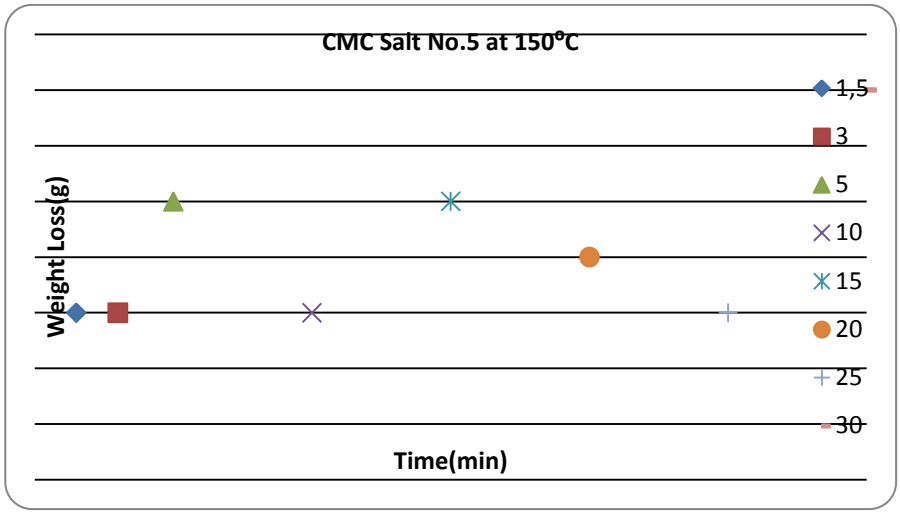

CMC Salt No.4 at 170°C
Weight Loss(g)
Time(min)

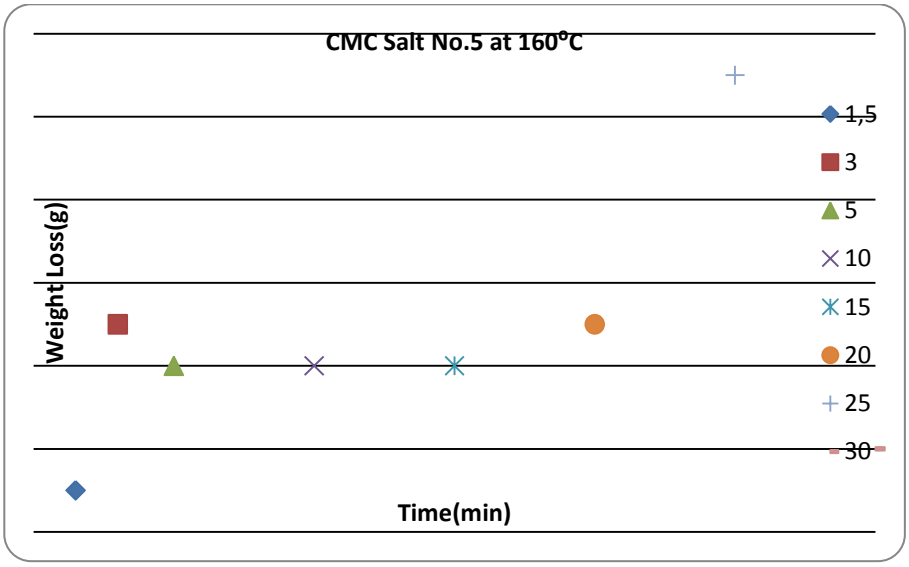

CMC Salt No.4 at 180°C
Weight Loss(g)
Time(min0
1,5
3
5
10
15
20
25
30

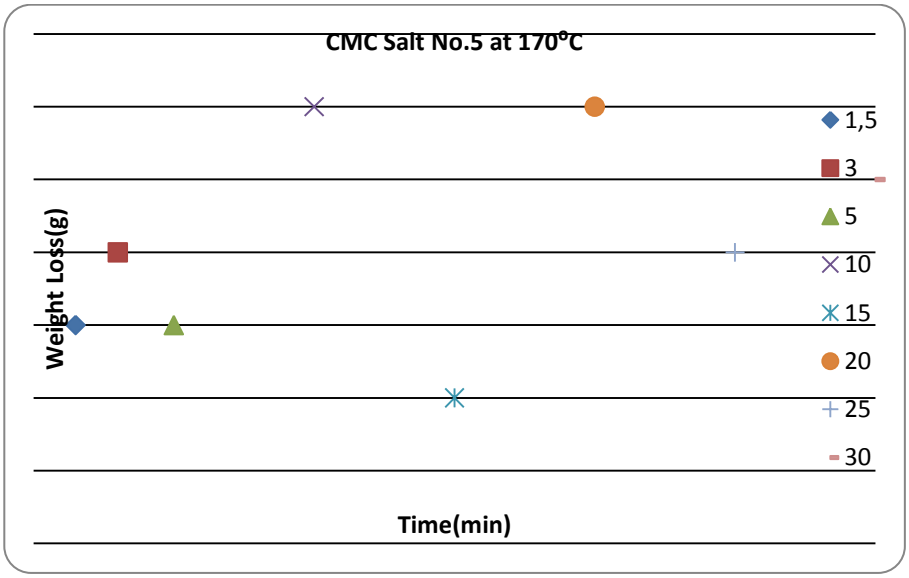


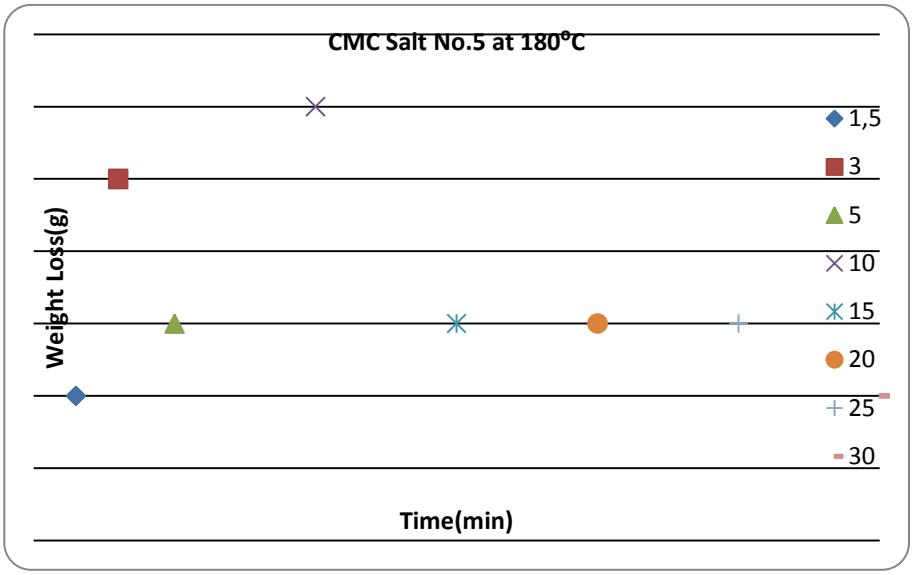


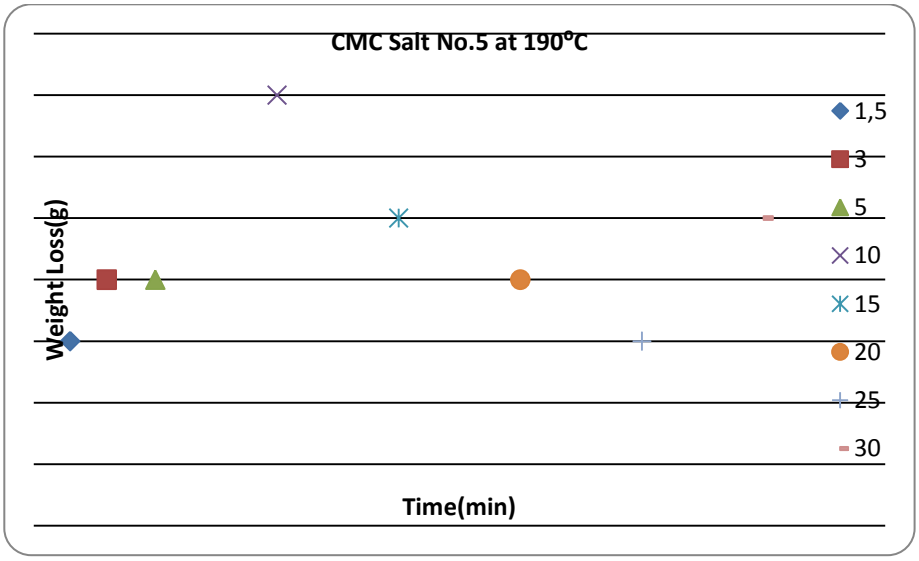
Graph of Weight Loss vs Time of samples that were crosslinked at different temperatures using salt No. 5:

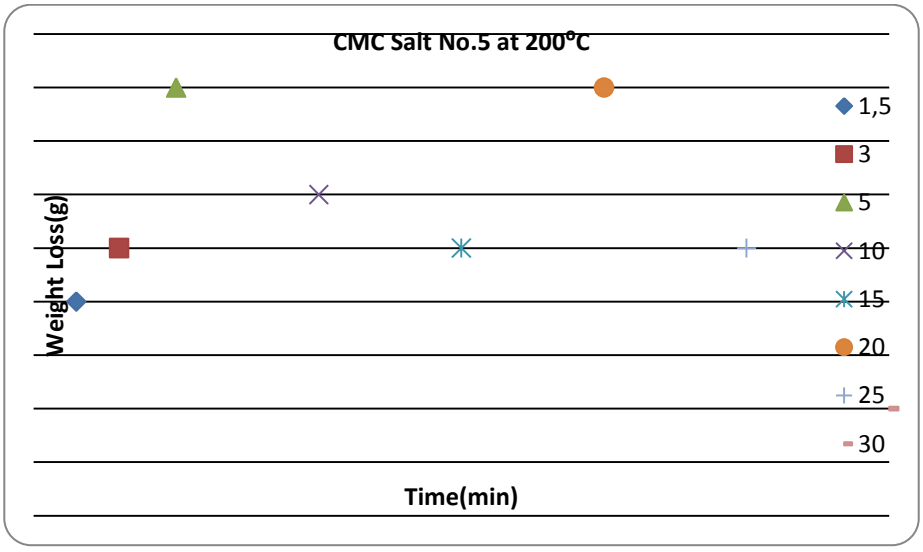

CMC Salt No.5 at 150°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30


CMC Salt No.5 at 160°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30


CMC Salt No.5 at 170°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30


CMC Salt No.5 at 180°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30


CMC Salt No.5 at 190°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30


CMC Salt No.5 at 200°C
Weight Loss(g)
Time(min)
1,5
3
5
10
15
20
25
30

Appendix 2

These tables show weights before and after crosslinking. Crosslinking was done at different temperatures and time.

Samples treated with CMC salt No. 1 crosslinked at 150°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	3	0.77	0.74	0.03
2	5	0.75	0.72	0.03
3	10	0.77	0.73	0.04
4	15	0.72	0.69	0.03
5	20	0.76	0.73	0.03
6	25	0.78	0.74	0.04
7	30	0.74	0.7	0.04

Samples treated with CMC salt No. 1 crosslinked at 160°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	3	0.73	0.7	0.03
2	5	0.8	0.75	0.05
3	10	0.78	0.74	0.04
4	15	0.75	0.71	0.04
5	20	0.76	0.71	0.05
6	25	0.78	0.74	0.04
7	30	0.77	0.72	0.05

Table 26: Samples treated with CMC salt No. 1 crosslinked at 170°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	3	0.77	0.71	0.06
2	5	0.77	0.7	0.07
3	10	0.8	0.73	0.07
4	15	0.77	0.7	0.07
5	20	0.78	0.71	0.07
6	25	0.78	0.69	0.09
7	30	0.79	0.72	0.07

Samples treated with CMC salt No. 1 crosslinked at 180°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST (g)
1	3	0.79	0.71	0.08
2	5	0.79	0.71	0.08
3	10	0.75	0.68	0.07
4	15	0.78	0.71	0.07
5	20	0.75	0.71	0.04
6	25	0.77	0.7	0.07
7	30	0.78	0.71	0.07

Samples treated with CMC salt No. 1 crosslinked at 190°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	3	0.78	0.74	0.04
2	5	0.74	0.7	0.04
3	10	0.76	0.71	0.05
4	15	0.68	0.62	0.06
5	20	0.67	0.62	0.05
6	25	0.79	0.72	0.07
7	30	0.78	0.7	0.08

Samples treated with CMC salt No. 1 crosslinked at 200°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	3	0.79	0.74	0.05
2	5	0.72	0.66	0.06
3	10	0.78	0.72	0.06
4	15	0.78	0.72	0.06
5	20	0.77	0.71	0.06
6	25	0.79	0.74	0.05
7	30	0.67	0.64	0.03

Samples treated with CMC salt No. 2 crosslinked at 150°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,15	0,15	0
2	3	0,19	0,18	0,01
3	5	0,22	0,2	0,02
4	10	0,22	0,19	0,03
5	15	0,26	0,23	0,03
6	20	0,28	0,27	0,01
7	25	0,23	0,21	0,02
8	30	0,17	0,16	0,01

Samples treated with CMC salt No. 2 crosslinked at 160°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,16	0,14	0,02
2	3	0,22	0,2	0,02
3	5	0,16	0,14	0,02
4	10	0,15	0,12	0,03
5	15	0,16	0,15	0,01
6	20	0,2	0,19	0,01
7	25	0,19	0,17	0,02
8	30	0,19	0,16	0,03

Samples treated with CMC salt No. 2 crosslinked at 170°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,19	0,17	0,02
2	3	0,18	0,17	0,01
3	5	0,25	0,22	0,03
4	10	0,23	0,2	0,03
5	15	0,22	0,19	0,03
6	20	0,15	0,14	0,01
7	25	0,2	0,17	0,03
8	30	0,21	0,17	0,04

Samples treated with CMC salt No. 2 crosslinked at 180°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST (g)
1	1,5	0,2	0,18	0,02
2	3	0,22	0,2	0,02
3	5	0,21	0,19	0,02
4	10	0,22	0,2	0,02
5	15	0,22	0,2	0,02
6	20	0,22	0,21	0,01
7	25	0,34	0,31	0,03
8	30	0,21	0,2	0,01

Samples treated with CMC salt No. 2 crosslinked at 190°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,3	0,29	0,01
2	3	0,23	0,22	0,01
3	5	0,26	0,24	0,02
4	10	0,27	0,25	0,02
5	15	0,23	0,21	0,02
6	20	0,27	0,25	0,02
7	25	0,28	0,25	0,03
8	30	0,25	0,24	0,01

Samples treated with CMC salt No. 2 crosslinked at 200°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,16	0,15	0,01
2	3	0,22	0,21	0,01
3	5	0,18	0,17	0,01
4	10	0,19	0,15	0,04
5	15	0,18	0,15	0,03
6	20	0,21	0,17	0,04
7	25	0,2	0,16	0,04
8	30	0,21	0,19	0,02

Samples treated with CMC salt No. 3 crosslinked at 150°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,38	0,36	0,02
2	3	0,34	0,31	0,03
3	5	0,38	0,34	0,04
4	10	0,32	0,29	0,03
5	15	0,34	0,31	0,03
6	20	0,32	0,29	0,03
7	25	0,4	0,36	0,04
8	30	0,31	0,29	0,02

Samples treated with CMC salt No. 3 crosslinked at 160°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,43	0,39	0,04
2	3	0,42	0,4	0,02
3	5	0,3	0,29	0,01
4	10	0,51	0,47	0,04
5	15	0,52	0,46	0,06
6	20	0,51	0,48	0,03
7	25	0,44	0,42	0,02
8	30	0,45	0,43	0,02

Samples treated with CMC salt No. 3 crosslinked at 170°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,28	0,28	0
2	3	0,3	0,28	0,02
3	5	0,38	0,33	0,05
4	10	0,59	0,53	0,06
5	15	0,51	0,47	0,04
6	20	0,49	0,43	0,06
7	25	0,52	0,48	0,04
8	30	0,37	0,35	0,02

Samples treated with CMC salt No. 3 crosslinked at 180°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST (g)
1	1,5	0,42	0,4	0,2
2	3	0,45	0,41	0,04
3	5	0,43	0,4	0,03
4	10	0,42	0,4	0,02
5	15	0,44	0,42	0,02
6	20	0,46	0,42	0,04
7	25	0,45	0,43	0,02
8	30	0,42	0,4	0,02

Samples treated with CMC salt No. 3 crosslinked at 190°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,4	0,38	0,02
2	3	0,33	0,31	0,02
3	5	0,23	0,21	0,02
4	10	0,25	0,25	0
5	15	0,33	0,28	0,05
6	20	0,38	0,33	0,05
7	25	0,33	0,3	0,03
8	30	0,45	0,43	0,02

Samples treated with CMC salt No. 3 crosslinked at 200°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,34	0,32	0,02
2	3	0,44	0,4	0,04
3	5	0,48	0,44	0,04
4	10	0,41	0,39	0,02
5	15	0,53	0,46	0,07
6	20	0,43	0,38	0,05
7	25	0,42	0,35	0,07
8	30	0,47	0,43	0,04

Samples treated with CMC salt No. 4 crosslinked at 150°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,28	0,27	0,01
2	3	0,24	0,24	0
3	5	0,26	0,26	0
4	10	0,28	0,26	0,02
5	15	0,3	0,27	0,03
6	20	0,3	0,26	0,04
7	25	0,3	0,28	0,02
8	30	0,27	0,25	0,02

Samples treated with CMC salt No. 4 crosslinked at 160°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,26	0,25	0,01
2	3	0,25	0,22	0,03
3	5	0,34	0,33	0,01
4	10	0,26	0,25	0,01
5	15	0,36	0,32	0,04
6	20	0,39	0,36	0,03
7	25	0,21	0,2	0,01
8	30	0,25	0,24	0,01

Samples treated with CMC salt No. 4 crosslinked at 170°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,27	0,26	0,1
2	3	0,21	0,2	0,01
3	5	0,26	0,21	0,05
4	10	0,39	0,35	0,04
5	15	0,22	0,21	0,01
6	20	0,24	0,22	0,02
7	25	0,26	0,25	0,01
8	30	0,18	0,17	0,01

Samples treated with CMC salt No. 4 crosslinked at 180°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST (g)
1	1,5	0,26	0,25	0,01
2	3	0,25	0,24	0,01
3	5	0,27	0,25	0,02
4	10	0,25	0,23	0,02
5	15	0,25	0,22	0,03
6	20	0,32	0,29	0,03
7	25	0,22	0,21	0,01
8	30	0,19	0,16	0,03

Samples treated with CMC salt No. 4 crosslinked at 190°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,24	0,23	0,01
2	3	0,24	0,2	0,02
3	5	0,27	0,24	0,03
4	10	0,23	0,23	0
5	15	0,28	0,24	0,04
6	20	0,26	0,24	0,02
7	25	0,28	0,26	0,02
8	30	0,28	0,26	0,02

Samples treated with CMC salt No. 4 crosslinked at 200°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,25	0,21	0,04
2	3	0,25	0,2	0,05
3	5	0,26	0,24	0,02
4	10	0,27	0,25	0,02
5	15	0,21	0,2	0,01
6	20	0,26	0,24	0,02
7	25	0,34	0,31	0,03
8	30	0,21	0,19	0,02

Samples treated with CMC salt No. 5 crosslinked at 150°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,16	0,16	0
2	3	0,21	0,21	0
3	5	0,18	0,17	0,01
4	10	0,15	0,13	0,02
5	15	0,19	0,18	0,01
6	20	0,18	0,16	0,02
7	25	0,18	0,17	0,01
8	30	0,13	0,12	0,01

Samples treated with CMC salt No. 5 crosslinked at 160°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,24	0,23	0,01
2	3	0,14	0,13	0,01
3	5	0,18	0,17	0,01
4	10	0,18	0,17	0,01
5	15	0,19	0,16	0,03
6	20	0,19	0,15	0,04
7	25	0,21	0,2	0,01
8	30	0,13	0,12	0,01

Samples treated with CMC salt No. 5 crosslinked at 170°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,09	0,07	0,02
2	3	0,13	0,11	0,02
3	5	0,15	0,11	0,04
4	10	0,17	0,16	0,01
5	15	0,29	0,25	0,04
6	20	0,2	0,16	0,04
7	25	0,23	0,18	0,05
8	30	0,11	0,09	0,02

Samples treated with CMC salt No. 5 crosslinked at 180°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST (g)
1	1,5	0,16	0,14	0,02
2	3	0,18	0,17	0,01
3	5	0,16	0,13	0,03
4	10	0,15	0,1	0,04
5	15	0,11	0,09	0,02
6	20	0,14	0,12	0,02
7	25	0,13	0,09	0,04
8	30	0,15	0,14	0,01

Samples treated with CMC salt No. 5 crosslinked at 190°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,11	0,08	0,03
2	3	0,08	0,07	0,01
3	5	0,16	0,15	0,01
4	10	0,14	0,13	0,02
5	15	0,15	0,14	0,01
6	20	0,15	0,14	0,01
7	25	0,11	0,1	0,01
8	30	0,12	0,11	0,01

Samples treated with CMC salt No. 5 crosslinked at 200°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,13	0,11	0,03
2	3	0,13	0,11	0,02
3	5	0,14	0,1	0,03
4	10	0,17	0,13	0,04
5	15	0,15	0,12	0,03
6	20	0,16	0,15	0,01
7	25	0,15	0,14	0,01
8	30	0,16	0,13	0,03

Samples treated with CMC salt No. 6 crosslinked at 150°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,55	0,52	0,03
2	3	0,43	0,4	0,03
3	5	0,42	0,37	0,05
4	10	0,43	0,4	0,03
5	15	0,44	0,39	0,05
6	20	0,47	0,43	0,04
7	25	0,46	0,43	0,03
8	30	0,43	0,36	0,07

Samples treated with CMC salt No. 6 crosslinked at 160°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,49	0,48	0,01
2	3	0,54	0,49	0,05
3	5	0,58	0,54	0,04
4	10	0,76	0,72	0,04
5	15	0,53	0,49	0,04
6	20	0,5	0,45	0,05
7	25	0,57	0,46	0,11
8	30	0,4	0,38	0,02

Samples treated with CMC salt No. 6 crosslinked at 170°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,5	0,47	0,03
2	3	0,5	0,46	0,04
3	5	49	0,46	0,03
4	10	0,45	0,39	0,06
5	15	0,52	0,5	0,02
6	20	0,45	0,39	0,06
7	25	0,53	0,49	0,04
8	30	0,64	0,59	0,05

Samples treated with CMC salt No. 6 crosslinked at 180°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST (g)
1	1,5	0,66	0,64	0,02
2	3	0,67	0,62	0,05
3	5	0,7	0,67	0,03
4	10	0,64	0,58	0,06
5	15	0,42	0,39	0,03
6	20	0,59	0,56	0,03
7	25	0,44	0,41	0,03
8	30	0,47	0,45	0,02

Samples treated with CMC salt No. 6 crosslinked at 190°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,46	0,43	0,03
2	3	0,72	0,68	0,04
3	5	0,43	0,39	0,04
4	10	0,6	0,53	0,07
5	15	0,48	0,43	0,05
6	20	0,48	0,44	0,04
7	25	0,41	0,39	0,03
8	30	0,45	0,4	0,05

Samples treated with CMC salt No. 6 crosslinked at 200°C

SAMPLE No.	TIME(min)	WEIGHT BEFORE(g)	WEIGHT AFTER(g)	WEIGHT LOST(g)
1	1,5	0,42	0,38	0,04
2	3	0,44	0,39	0,05
3	5	0,62	0,54	0,08
4	10	0,48	0,42	0,06
5	15	0,45	0,4	0,05
6	20	0,51	0,43	0,08
7	25	0,39	0,34	0,05
8	30	0,34	0,32	0,02

Appendix 3

These tables show weights before and after immersing to water. Immersing was done in various of time. After that were samples dryed for 1 hour and half at 105 °C.

Immersing of Samples treated with CMC salt No. 1 crosslinked at 150°C

Samples crosslinked at 150°C SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105°C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0.2	0.16	2.32
2	0.19	0.15	1.77
3	0.19	0.16	2.57
4	0.2	0.18	3.66
5	0.2	0.15	3.34
6	0.25	0.2	3.98
7	0.22	0.19	4.23
8	0.25	0.21	5.09

Immersing of Samples treated with CMC salt No. 1 crosslinked at 160°C

SAMPLE crosslinked at 160°C Sample No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105°C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0.23	0.16	2.83
2	0.19	0.17	2.31
3	0.23	0.21	2.51
4	0.25	0.23	3.86
5	0.17	0.15	2.94
6	0.22	0.19	3.3
7	0.2	0.18	4.42
8	0.21	0.2	2.21

Immersing of Samples treated with CMC salt No. 1 crosslinked at 170°C

SAMPLE crosslinked at 170°C Sample No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0.17	0.15	2.38
2	0.21	0.18	5.05
3	0.16	0.14	3.89
4	0.22	0.19	5.44
5	0.19	0.16	4.25
6	0.25	0.22	5.62
7	0.24	0.2	4.98
8	0.21	0.19	4.28

Immersing of Samples treated with CMC salt No. 1 crosslinked at 180°C

SAMPLES crosslinked at 180^{0}C Sample No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0.26	0.22	2.59
2	0.21	0.18	4.33
3	0.25	0.2	5
4	0.23	0.19	5.02
5	0.2	0.18	4.56
6	0.18	0.14	3.35
7	0.18	0.16	3.33
8	0.21	0.17	3.62

Immersing of Samples treated with CMC salt No. 1 crosslinked at 190°C

SAMPLES crosslinked at 190^{0}C Sample No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0.16	0.15	2.19
2	0.25	0.22	4.86
3	0.22	0.21	5.39
4	0.19	0.17	5.39
5	0.2	0.18	4.86
6	0.23	0.21	2.84
7	0.2	0.18	3.43
8	0.19	0.17	3.32

Immersing of Samples treated with CMC salt No. 1 crosslinked at 200°C

SAMPLES Crosslinked at 200^0C Sample No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^0C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0.19	0.17	2.84
2	0.19	0.16	4.42
3	0.24	0.22	5.33
4	0.24	0.22	3.71
5	0.23	0.21	2.59
6	0.19	0.17	2.12
7	0.17	0.13	1.7
8	0.22	0.18	2.16

Immersing of Samples treated with CMC salt No. 2 crosslinked at 150°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^0C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,13	0,09	1,23
2	0,12	0,1	1,75
3	0,15	0,13	1,91
4	0,11	0,09	1,79
5	0,15	0,13	2,57
6	0,18	0,15	2,94
7	0,17	0,15	2,83
8	0,17	0,15	3,09

Immersing of Samples treated with CMC salt No. 2 crosslinked at 160°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^0C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,12	0,1	1,36
2	0,15	0,12	1,85
3	0,09	0,08	1,4
4	0,11	0,1	1,92
5	0,15	0,1	2,57
6	0,13	0,11	2,96
7	0,15	0,11	2,53
8	0,14	0,11	2,77

Immersing of Samples treated with CMC salt No. 2 crosslinked at 170°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,13	0,11	1,77
2	0,16	0,12	2,77
3	0,23	0,18	3,16
4	0,17	0,14	3,45
5	0,17	0,13	3,07
6	0,07	0,05	2,38
7	0,14	0,12	2,98
8	0,14	0,12	3,19

Immersing of Samples treated with CMC salt No. 2 crosslinked at 180°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,12	0,11	1,72
2	0,12	0,1	2,88
3	0,11	0,1	3,14
4	0,16	0,15	3,02
5	0,16	0,15	3,42
6	0,19	0,14	3,72
7	0,24	0,21	3,62
8	0,17	0,16	3,11

Immersing of Samples treated with CMC salt No. 2 crosslinked at 190°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,2	0,17	3,08
2	0,16	0,13	3,34
3	0,19	0,16	3,64
4	0,21	0,16	2,52
5	0,16	0,15	2,1
6	0,2	0,16	2,1
7	0,13	0,12	1,96
8	0,16	0,14	1,8

Immersing of Samples treated with CMC salt No. 2 crosslinked at 200°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,1	0,08	3,53
2	0,17	0,14	3,78
3	0,14	0,1	2,31
4	0,11	0,09	1,65
5	0,11	0,08	1,29
6	0,11	0,09	1,25
7	0,11	0,09	1,24
8	0,14	0,12	1,55

Immersing of Samples treated with CMC salt No. 3 crosslinked at 150°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,12	0,09	1,28
2	0,11	0,07	1,39
3	0,13	0,12	1,76
4	0,12	0,11	1,81
5	0,08	0,08	1,17
6	0,1	0,1	1,1
7	0,14	0,14	1,53
8	0,13	0,11	1,4

Immersing of Samples treated with CMC salt No. 3 crosslinked at 160°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,15	0,14	1,4
2	0,14	0,11	1,35
3	0,08	0,05	1,28
4	0,11	0,1	1,67
5	0,11	0,09	1,54
6	0,13	0,11	1,64
7	0,15	0,09	1,69
8	0,19	0,14	1,79

Immersing of Samples treated with CMC salt No. 3 crosslinked at 170°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,1	0,09	1,35
2	0,11	0,08	1,38
3	0,17	0,16	1,76
4	0,19	0,18	1,78
5	0,14	0,13	1,3
6	0,22	0,21	2,38
7	0,16	0,14	2,17
8	0,11	0,1	1,47

Immersing of Samples treated with CMC salt No. 3 crosslinked at 180°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,19	0,18	1,89
2	0,21	0,16	1,88
3	0,19	0,15	2,1
4	0,21	0,17	2,29
5	0,18	0,17	2,75
6	0,16	0,16	2,36
7	0,14	0,14	3,39
8	0,26	0,21	3,71

Immersing of Samples treated with CMC salt No. 3 crosslinked at 190°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,15	0,14	1,47
2	0,17	0,16	2,19
3	0,11	0,09	1,69
4	0,1	0,08	1,69
5	0,15	0,14	5,01
6	0,2	0,19	5,06
7	0,11	0,09	3,73
8	0,19	0,18	5,89

Immersing of Samples treated with CMC salt No. 3 crosslinked at 200°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,15	0,14	1,64
2	0,2	0,19	2,89
3	0,15	0,14	2
4	0,18	0,16	4,5
5	0,2	0,14	4,47
6	0,16	0,12	2,37
7	0,09	0,07	5,69
8	0,27	0,25	5,03

Immersing of Samples treated with CMC salt No. 4 crosslinked at 150°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,16	0,15	1,92
2	0,15	0,14	1,7
3	0,17	0,16	1,93
4	0,16	0,15	2,3
5	0,17	0,16	2,61
6	0,15	0,14	2,51
7	0,16	0,15	2,89
8	0,18	0,16	2,81

Immersing of Samples treated with CMC salt No. 4 crosslinked at 160°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,14	0,12	1,15
2	0,16	0,14	1,46
3	0,22	0,2	1,75
4	0,18	0,15	2,02
5	0,19	0,18	2,63
6	0,23	0,2	2,76
7	0,12	0,1	2,36
8	0,22	0,17	3,96

Immersing of Samples treated with CMC salt No. 4 crosslinked at 170°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,19	0,14	2,26
2	0,13	0,11	2,6
3	0,13	0,11	2,91
4	0,24	0,16	3,74
5	0,11	0,06	2,27
6	0,15	0,14	2,97
7	0,14	0,13	3,12
8	0,1	0,09	3,02

Immersing of Samples treated with CMC salt No. 4 crosslinked at 180°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,19	0,18	2,87
2	0,15	0,13	2,72
3	0,16	0,14	3,15
4	0,15	0,13	2,36
5	0,17	0,16	2,98
6	0,17	0,16	2,74
7	0,1	0,08	1,86
8	0,16	0,14	1,99

Immersing of Samples treated with CMC salt No. 4 crosslinked at 190°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,22	0,14	3,3
2	0,13	0,11	2,35
3	0,1	0,09	2,91
4	0,14	0,12	3,06
5	0,14	0,13	2,46
6	0,16	0,15	2,41
7	0,15	0,14	2,17
8	0,19	0,18	2,95

Immersing of Samples treated with CMC salt No. 4 crosslinked at 200°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,17	0,1	2,98
2	0,16	0,14	3,17
3	0,18	0,17	3,16
4	0,15	0,13	2,68
5	0,13	0,12	1,79
6	0,14	0,12	1,96
7	0,16	0,14	1,88
8	0,13	0,12	1,54

Immersing of Samples treated with CMC salt No. 5 crosslinked at 150°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,11	0,05	2,74
2	0,09	0,06	2,7
3	0,13	0,07	2,89
4	0,1	0,06	2,23
5	0,09	0,05	2,33
6	0,09	0,05	2,54
7	0,09	0,08	2,47
8	0,14	0,1	2,77

Immersing of Samples treated with CMC salt No. 5 crosslinked at 160°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,13	0,13	3,99
2	0,1	0,08	2,85
3	0,09	0,08	3,46
4	0,1	0,09	3,03
5	0,12	0,1	2,52
6	0,1	0,09	2,2
7	0,09	0,08	2,69
8	0,11	0,1	1,41

Immersing of Samples treated with CMC salt No. 5 crosslinked at 170°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,1	0,09	2,87
2	0,09	0,08	2,56
3	0,08	0,07	1,99
4	0,11	0,1	2,25
5	0,18	0,16	1,7
6	0,09	0,08	1,2
7	0,12	0,1	0,93
8	0,08	0,07	0,92

Immersing of Samples treated with CMC salt No. 5 crosslinked at 180°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,1	0,09	1,06
2	0,11	0,09	2,37
3	0,1	0,09	2,34
4	0,09	0,08	1,13
5	0,07	0,06	0,81
6	0,09	0,08	0,71
7	0,07	0,06	0,66
8	0,1	0,09	3,02

Immersing of Samples treated with CMC salt No. 5 crosslinked at 190°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,1	0,08	2,97
2	0,06	0,04	1,6
3	0,11	0,1	1,84
4	0,08	0,05	0,66
5	0,09	0,07	0,71
6	0,09	0,08	0,65
7	0,09	0,06	0,52
8	0,09	0,07	0,58

Immersing of Samples treated with CMC salt No. 5 crosslinked at 200°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,08	0,04	1,72
2	0,08	0,05	1,02
3	0,1	0,06	1,32
4	0,11	0,07	0,99
5	0,1	0,05	0,66
6	0,12	0,05	0,68
7	0,1	0,08	0,56
8	0,1	0,08	0,54

Immersing of Samples treated with CMC salt No. 6 crosslinked at 150°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,21	0,19	1,24
2	0,14	0,14	1,58
3	0,18	0,18	1,58
4	0,16	0,15	1,25
5	0,16	0,16	1,66
6	0,2	0,2	1,84
7	0,23	0,2	2,18
8	0,19	0,15	1,81

Immersing of Samples treated with CMC salt No. 6 crosslinked at 160°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,16	0,15	1,76
2	0,23	0,19	1,49
3	0,21	0,14	2,11
4	0,25	0,23	2,07
5	0,18	0,16	1,75
6	0,19	0,15	2,02
7	0,21	0,2	2,43
8	0,18	0,18	2,34

Immersing of Samples treated with CMC salt No. 6 crosslinked at 170°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,21	0,19	1,31
2	0,2	0,2	1,52
3	0,11	0,17	2,17
4	0,15	0,15	1,9
5	0,14	0,15	1,41
6	0,12	0,12	1,92
7	0,13	0,08	1,99
8	0,14	0,14	2,46

Immersing of Samples treated with CMC salt No. 6 crosslinked at 180°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,19	0,18	2,02
2	0,22	0,21	2,09
3	0,2	0,19	1,99
4	0,15	0,12	2,42
5	0,13	0,13	2,61
6	0,19	0,14	2,88
7	0,14	0,14	3,59
8	0,11	0,1	3,53

Immersing of Samples treated with CMC salt No. 6 crosslinked at 190°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,14	0,13	1,42
2	0,2	0,19	2,24
3	0,13	0,14	2,13
4	0,18	0,17	3,37
5	0,14	0,13	4,09
6	0,14	0,11	3,29
7	0,17	0,15	3,91
8	0,13	0,13	3,22

Immersing of Samples treated with CMC salt No. 6 crosslinked at 200°C

SAMPLE No.	WEIGHT OF SAMPLE(g) BEFORE DRYING	WEIGHT OF SAMPLE(g) AFTER DRYING @105^{0}C FOR 1HR 30 MIN	WEIGHT OF SAMPLE AFTER IMMERSING IN WATER FOR 1HR
1	0,13	0,13	1,66
2	0,14	0,13	2,35
3	0,11	0,11	2,87
4	0,18	0,18	3,81
5	0,13	0,13	2,83
6	0,14	0,13	1,92
7	0,09	0,08	1,6
8	0,13	0,1	1,89